

Supporting Information

Visible-light-induced bromine radical initiate direct C–H alkylation of heteroaromatic

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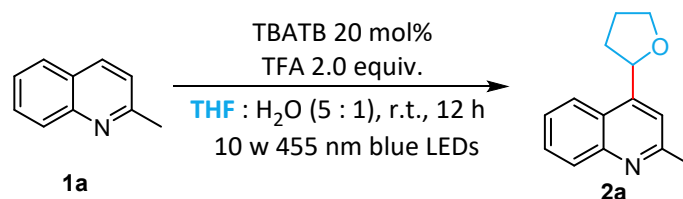
1. General considerations

General. Unless otherwise noted, all reactions were carried out under an air atmosphere. Analytical thin-layer chromatography (TLC) was performed on glass plates coated with 0.25 mm 230–400 mesh silica gel containing a fluorescent indicator. Visualization was accomplished by exposure to a UV lamp. All the products in this article are compatible with standard silica gel chromatography. Column chromatography was performed on silica gel (200–300 mesh). Eluent generally contained ethyl acetate (EA), petroleum ether (PE).

Structural analysis. NMR spectra were measured on a Bruker Ascend 400 spectrometer and chemical shifts (δ) are reported in parts per million (ppm). ^1H NMR spectra were recorded at 400 MHz in NMR solvents and referenced internally to corresponding solvent resonance, ^{13}C NMR spectra were recorded at 101 MHz, ^{19}F NMR spectra were recorded at 376 MHz, and referenced to corresponding solvent resonance. Coupling constants are reported in Hz with multiplicities denoted as s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet) and br (broad). Infrared spectra were collected on a Thermo Fisher Nicolet 6700 FT-IR spectrometer using ATR (Attenuated Total Reflectance) method. Absorption maxima (ν max) are reported in wavenumbers (cm^{-1}). High resolution mass spectra (HRMS) were acquired on Thermo Scientific LTQ Orbitrap XL with an ESI source. Melting points were measured with a micro-melting point apparatus.

Materials. Commercial reagents and solvent were purchased from Adamas, J&K, Energy, Aladdin, Alfa Aesar, Macklin, Organics, TCI, Innochem and used as received unless otherwise stated.

2. General procedure



A flame-dried 25 mL quartz reaction tube was placed with a magnetic stir bar. Then, 2-methylquinoline (28.6 mg, 0.2 mmol, 1.0 equiv.), tetrabutylammonium tribromide (19.3 mg, 0.04 mmol, 20.0 mol%), trifluoroacetic acid (45.6 mg, 0.4 mmol, 2.0 equiv.), THF (2.0 mL) and H₂O (400 μ L) were added to the tube. The reaction tube was placed on a photocatalytic parallel reactor with Blue LEDs (10 W) at the bottom (**Figure S1**). Then the reaction mixture was stirred and irradiated with the Blue LEDs for 12 hours at room temperature.

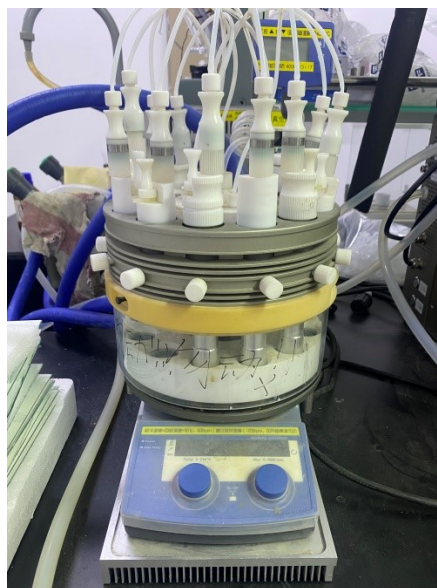


Figure S1. Picture of the reactor

After taking the reaction tube out, 10 mL saturated NaHCO₃ solution was added to the reaction mixture. Then, the reaction mixture was extracted with ethyl acetate (3 \times 10 mL). The combined organic phase was washed with brine (2 \times 5.0 mL) and then dried over anhydrous Na₂SO₄. After concentration, the crude product was purified by column chromatography (silica gel) to give the target product, using petroleum ether / ethyl acetate as the eluent.

3. Radical inhibition experiment

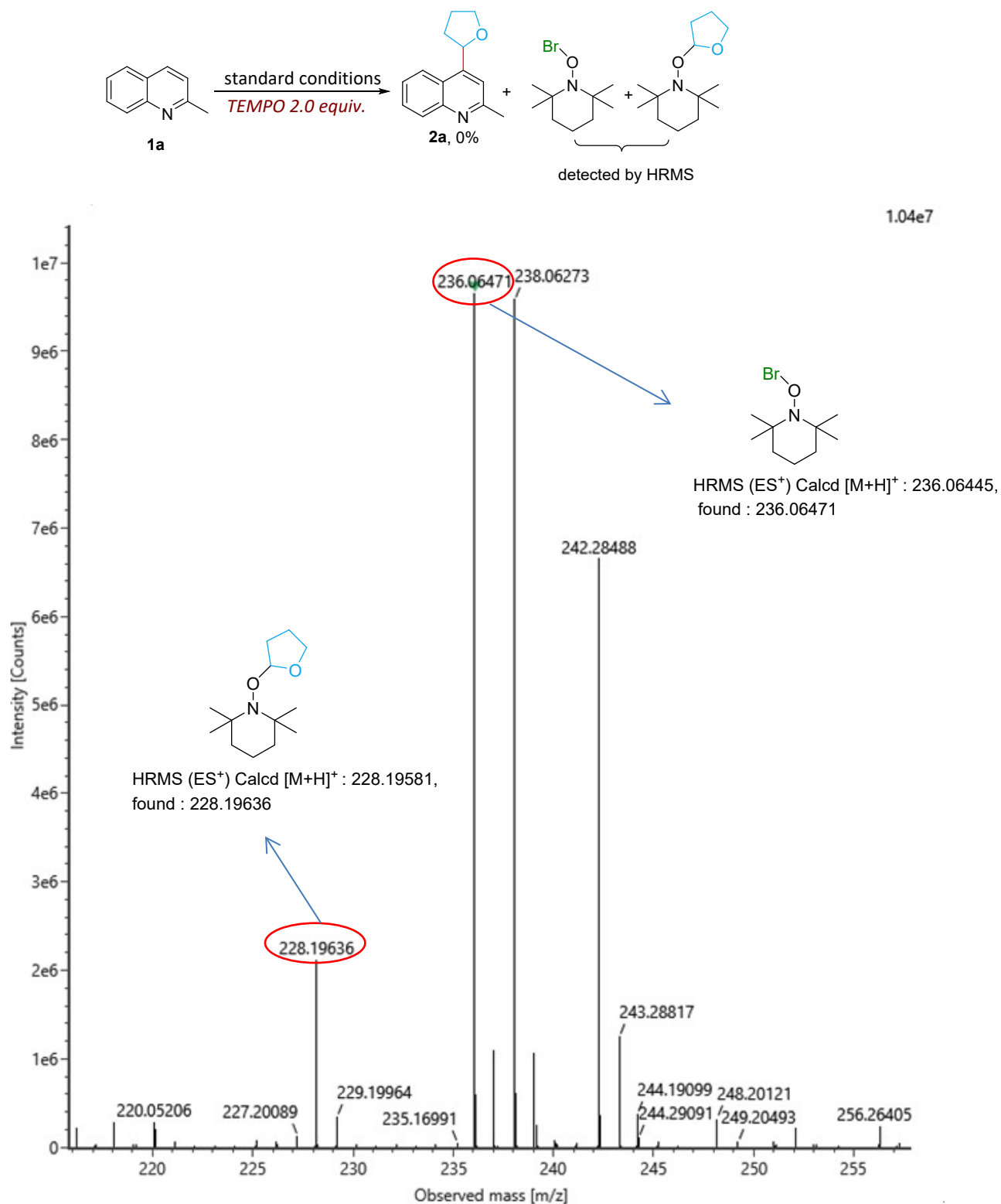
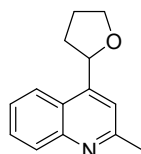


Figure S2. HRMS spectra for Radical inhibition experiment.

4. Characterization data

(2a) 2-Methyl-4-(tetrahydro-2-furanyl)quinolone (CAS: 104293-35-8)¹



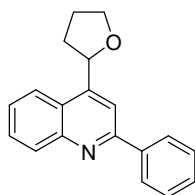
2-methyl-4-(tetrahydrofuran-2-yl)quinolone
Chemical Formula: C₁₄H₁₅NO
Exact Mass: 213.1154
Molecular Weight: 213.2800

Following the General Procedure A with 2-methylquinoline (28.6 mg, 0.2 mmol), **2a** was obtained as colorless liquid (36.3 mg, 85%), $R_f = 0.4$ (petroleum ether/ethyl acetate = 4:1).

¹H NMR (400 MHz, CDCl₃) δ 8.05 (d, $J = 8.4$ Hz, 1H), 7.84 (d, $J = 8.4$ Hz, 1H), 7.66 (t, $J = 8.0$ Hz, 1H), 7.49 – 7.44 (m, 2H), 5.58 – 5.55 (m, 1H), 4.25 – 4.21 (m, 1H), 4.06 – 4.01 (m, 1H), 2.74 (s, 3H), 2.64 – 2.56 (m, 1H), 2.11 – 1.96 (m, 2H), 1.87 – 1.79 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 159.0, 149.4, 147.8, 129.3, 128.9, 125.5, 123.8, 122.9, 117.2, 76.7, 68.9, 33.8, 25.9, 25.4.

(2b) 2-Phenyl-4-(tetrahydro-2-furanyl)quinolone (1869978-48-2)²



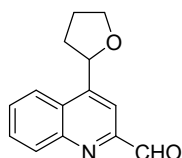
2-phenyl-4-(tetrahydrofuran-2-yl)quinolone
Chemical Formula: C₁₉H₁₇NO
Exact Mass: 275.1310
Molecular Weight: 275.3510

Following the General Procedure A with 2-phenylquinoline (41.0 mg, 0.2 mmol), **2b** was obtained as colorless oil (40.2 mg, 73%), $R_f = 0.4$ (petroleum ether/ethyl acetate = 4:1).

¹H NMR (400 MHz, CDCl₃) δ 8.24 – 8.20 (m, 3H), 8.06 (s, 1H), 7.90 (d, $J = 8.4$ Hz, 1H), 7.74 – 7.70 (m, 1H), 7.56 – 7.52 (m, 3H), 7.49 – 7.45 (m, 1H), 5.68 – 5.64 (m, 1H), 4.31 – 4.25 (m, 1H), 4.11 – 4.05 (m, 1H), 2.70 – 2.61 (m, 1H), 2.13 – 1.98 (m, 2H), 1.94 – 1.86 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 157.3, 149.9, 148.3, 139.9, 130.5, 129.2, 129.1, 128.7, 127.6, 126.0, 124.5, 123.0, 114.3, 69.0, 34.0, 26.0.

(2c) 4-(Tetrahydrofuran-2-yl)quinoline-2-carbaldehyde



4-(tetrahydrofuran-2-yl)quinoline-2-carbaldehyde
Chemical Formula: C₁₄H₁₃NO₂
Exact Mass: 227.0946
Molecular Weight: 227.2630

Following the General Procedure A with quinoline-2-carbaldehyde (31.4 mg, 0.2 mmol), **2c** was obtained as white solid (15.5 mg, 34%), $R_f = 0.3$ (petroleum ether/ethyl acetate = 5:1).

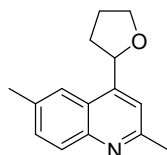
¹H NMR (400 MHz, CDCl₃) δ 10.22 (s, 1H), 8.27 (d, $J = 8.4$ Hz, 1H), 8.18 (s, 1H), 8.00 (d, $J = 8.8$ Hz, 1H), 7.83 – 7.79 (m, 1H), 7.71 – 7.67 (m, 1H), 5.66 – 5.62 (m, 1H), 4.30 – 4.25 (m, 1H), 4.10 – 4.04 (m, 1H), 2.68 – 2.60 (m, 1H), 2.10 – 2.02 (m, 2H), 1.91 – 1.83 (m, 1H).

¹³C NMR 101 MHz, CDCl₃) δ 194.0, 152.5, 151.3, 148.0, 131.3, 130.0, 128.9, 127.6, 127.4, 123.5, 112.9, 69.1, 33.9, 26.0.

Melting point (°C): 100.3 – 102.7 °C.

IR: 3375, 3059, 2988, 2955, 2878, 1697, 1593, 1512, 1458, 1360, 1151, 771, 650, 461.
HRMS (ESI) m/z calcd for $C_{14}H_{13}NO_2$ $[M+H]^+$: 228.10191, found: 228.10185.

(2d) 2,6-Dimethyl-4-(tetrahydrofuran-2-yl)quinolone (2378441-92-8)³



2,6-dimethyl-4-
(tetrahydrofuran-2-yl)quinolone
Chemical Formula: $C_{15}H_{17}NO$
Exact Mass: 227.1310
Molecular Weight: 227.3070

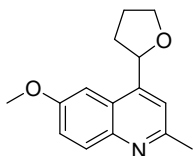
Following the General Procedure A with 2,6-dimethylquinoline (31.4 mg, 0.2 mmol), **2d** was obtained as colorless oil (20.0 mg, 44%), $R_f = 0.2$ (petroleum ether/ethyl acetate = 4:1).

1H NMR (400 MHz, $CDCl_3$) δ = 7.94 (d, $J = 8.4$ Hz, 1H), 7.58 (s, 1H), 7.49 (d, $J = 8.4$ Hz, 1H), 7.40 (s, 1H), 5.54 (t, $J = 7.2$ Hz, 1H), 4.24 – 4.19 (m, 1H), 4.06 – 4.00 (m, 1H), 2.71 (s, 3H), 2.63 – 2.56 (m, 1H), 2.52 (s, 3H), 2.12 – 1.95 (m,

2H), 1.87 – 1.78 (m, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 158.0, 148.7, 146.3, 135.2, 131.1, 129.0, 123.8, 122.0, 117.1, 76.7, 68.9, 33.8, 25.9, 25.3, 21.8.

(2e) 6-Methoxy-2-methyl-4-(tetrahydrofuran-2-yl)quinolone (2378441-93-9)⁴



6-methoxy-2-methyl-4-
(tetrahydrofuran-2-yl)quinolone
Chemical Formula: $C_{15}H_{17}NO_2$
Exact Mass: 243.1259
Molecular Weight: 243.3060

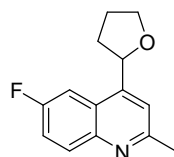
Following the General Procedure A with 6-methoxy-2-methylquinoline (34.6 mg, 0.2 mmol), **2e** was obtained as white solid (33.6 mg, 69%), $R_f = 0.1$ (petroleum ether/ethyl acetate = 4:1).

1H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, $J = 9.2$ Hz, 1H), 7.40 (s, 1H), 7.33 (dd, $J = 9.2, 2.4$ Hz, 1H), 7.08 (d, $J = 2.8$ Hz, 1H), 5.48 (t, $J = 7.2$ Hz, 1H), 4.25 – 4.19 (m, 1H), 4.06 – 4.00 (m, 1H), 3.91 (s, 3H), 2.70 (s, 3H), 2.63 – 2.55 (m, 1H),

2.11 – 2.05 (m, 1H), 2.04 – 1.96 (m, 1H), 1.89 – 1.82 (m, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 156.9, 156.3, 148.0, 143.7, 130.6, 124.6, 120.6, 117.4, 102.0, 76.8, 68.9, 55.5, 33.4, 25.9, 25.1.

(2f) 6-Fluoro-2-methyl-4-(tetrahydrofuran-2-yl)quinolone (2095358-07-7)⁴



6-fluoro-2-methyl-4-
(tetrahydrofuran-2-yl)quinolone
Chemical Formula: $C_{14}H_{14}FNO$
Exact Mass: 231.1059
Molecular Weight: 231.2704

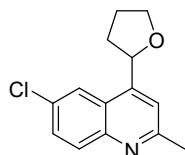
Following the General Procedure A with 6-fluoro-2-methylquinoline (32.2 mg, 0.2 mmol), **2f** was obtained as white solid (24.5 mg, 53%), $R_f = 0.3$ (petroleum ether/ethyl acetate = 5:1).

1H NMR (600 MHz, $CDCl_3$) δ 8.02 (dd, $J = 8.8, 5.6$ Hz, 1H), 7.45 – 7.39 (m, 3H), 5.42 (t, $J = 7.2$ Hz, 1H), 4.23 – 4.18 (m, 1H), 4.05 – 3.99 (m, 1H), 2.70 (s, 3H), 2.61 – 2.53 (m, 1H), 2.12 – 2.04 (m, 1H), 2.03 – 1.94 (m, 1H), 1.85 – 1.76 (m,

1H).

^{13}C NMR (101 MHz, CDCl_3) δ 159.7 (d, $J = 247.2$ Hz), 158.3 (d, $J = 2.7$ Hz), 148.6 (d, $J = 5.5$ Hz), 145.0, 131.7 (d, $J = 9.2$ Hz), 124.4 (d, $J = 9.3$ Hz), 118.8 (d, $J = 25.5$ Hz), 117.9, 106.8 (d, $J = 22.6$ Hz), 76.7, 68.9, 33.6, 25.9, 25.3.
 ^{19}F NMR (376 MHz, CDCl_3) δ -113.8.

(2g) 6-Chloro-2-methyl-4-(tetrahydrofuran-2-yl)quinolone (2378441-89-3)⁴

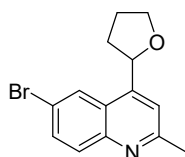


6-chloro-2-methyl-4-(tetrahydrofuran-2-yl)quinoline
Chemical Formula: $\text{C}_{14}\text{H}_{14}\text{ClNO}$
Exact Mass: 247.0764
Molecular Weight: 247.7220

Following the General Procedure A with 6-chloro-2-methylquinoline (35.5 mg, 0.2 mmol), **2g** was obtained as white solid (34.7 mg, 70%), $R_f = 0.4$ (petroleum ether/ethyl acetate = 4:1).

^1H NMR (400 MHz, CDCl_3) δ 7.96 (d, $J = 9.2$ Hz, 1H), 7.78 (d, $J = 2.0$ Hz, 1H), 7.58 (dd, $J = 8.8, 2.0$ Hz, 1H), 7.44 (s, 1H), 5.45 (t, $J = 7.2$ Hz, 1H), 4.20 (dd, $J = 13.6, 7.6$ Hz, 1H), 4.03 (dd, $J = 14.8, 7.2$ Hz, 1H), 2.71 (s, 3H), 2.64 – 2.55 (m, 1H), 2.14 – 2.05 (m, 1H), 2.04 – 1.95 (m, 1H), 1.85 – 1.76 (m, 1H).
 ^{13}C NMR (101 MHz, CDCl_3) δ 159.4, 148.5, 146.2, 131.2, 130.9, 129.7, 124.6, 122.1, 118.0, 76.5, 68.9, 33.8, 25.9, 25.4.

(2h) 6-Bromo-2-methyl-4-(tetrahydrofuran-2-yl)quinolone (2095358-06-6)⁴

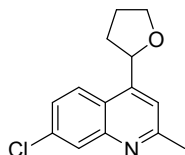


6-bromo-2-methyl-4-(tetrahydrofuran-2-yl)quinoline
Chemical Formula: $\text{C}_{14}\text{H}_{14}\text{BrNO}$
Exact Mass: 291.0259
Molecular Weight: 292.1760

Following the General Procedure A with 6-bromo-2-methylquinoline (44.4 mg, 0.2 mmol), **2h** was obtained as white solid (43.8 mg, 75%), $R_f = 0.2$ (petroleum ether/ethyl acetate = 4:1).

^1H NMR (400 MHz, CDCl_3) δ 7.97 (d, $J = 2.0$ Hz, 1H), 7.89 (d, $J = 9.2$ Hz, 1H), 7.71 (dd, $J = 8.8, 2.0$ Hz, 1H), 7.44 (s, 1H), 5.45 (t, $J = 7.2$ Hz, 1H), 4.23–4.17 (m, 1H), 4.02 (dd, $J = 15.2, 7.2$ Hz, 1H), 2.70 (s, 3H), 2.64 – 2.54 (m, 1H), 2.13 – 2.05 (m, 1H), 2.03 – 1.95 (m, 1H), 1.85 – 1.76 (m, 1H).
 ^{13}C NMR (101 MHz, CDCl_3) δ 159.5, 148.5, 146.4, 132.3, 131.0, 125.4, 125.1, 119.4, 118.0, 76.5, 68.9, 33.8, 25.9, 25.4.

(2i) 7-Chloro-2-methyl-4-(tetrahydrofuran-2-yl)quinolone (1821239-62-6)⁴



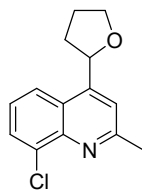
7-chloro-2-methyl-4-(tetrahydrofuran-2-yl)quinoline
Chemical Formula: $\text{C}_{14}\text{H}_{14}\text{ClNO}$
Exact Mass: 247.0764
Molecular Weight: 247.7220

Following the General Procedure A with 7-chloro-2-methylquinoline (35.5 mg, 0.2 mmol), **2i** was obtained as white solid (26.3 mg, 53%), $R_f = 0.4$ (petroleum ether/ethyl acetate = 4:1).

^1H NMR (400 MHz, CDCl_3) δ 8.03 (d, $J = 1.6$ Hz, 1H), 7.77 (d, $J = 8.8$ Hz, 1H), 7.43 – 7.40 (m, 2H), 5.49 (t, $J = 7.2$ Hz, 1H), 4.20 (dd, $J = 14.0, 8.0$ Hz, 1H), 4.02 (dd, $J = 15.2, 7.2$ Hz, 1H), 2.71 (s, 3H), 2.61 – 2.53 (m, 1H), 2.12 – 2.04 (m, 1H), 2.03 – 1.95 (m, 1H), 1.84 – 1.75 (m, 1H).

^{13}C NMR (101 MHz, CDCl_3) δ 160.3, 149.4, 148.4, 134.8, 128.3, 126.3, 124.3, 122.3, 117.4, 76.6, 69.0, 33.8, 25.9, 25.5.

(2j) 8-Chloro-2-methyl-4-(tetrahydrofuran-2-yl)quinolone (2378441-96-2)⁴



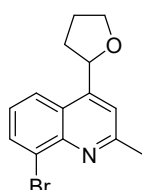
8-chloro-2-methyl-4-(tetrahydrofuran-2-yl)quinolone
Chemical Formula: $\text{C}_{14}\text{H}_{14}\text{ClNO}$
Exact Mass: 247.0764
Molecular Weight: 247.7220

Following the General Procedure A with 8-chloro-2-methylquinoline (35.5 mg, 0.2 mmol), **2j** was obtained as white solid (27.2 mg, 55%), $R_f = 0.5$ (petroleum ether/ethyl acetate = 5:1).

^1H NMR (400 MHz, CDCl_3) δ 7.80 – 7.76 (m, 2H), 7.51 (s, 1H), 7.39 (t, $J = 8.0$ Hz, 1H), 5.54 (t, $J = 7.2$ Hz, 1H), 4.23 (q, $J = 7.6$ Hz, 1H), 4.04 (q, $J = 7.2$ Hz, 1H), 2.82 (s, 3H), 2.64 – 2.56 (m, 1H), 2.13 – 2.05 (m, 1H), 2.04 – 1.95 (m, 1H), 1.86 – 1.77 (m, 1H).

^{13}C NMR (101 MHz, CDCl_3) δ 160.2, 149.7, 144.2, 133.5, 129.1, 125.3, 125.2, 122.1, 118.1, 76.7, 69.0, 33.9, 25.93, 25.91.

(2k) 8-Bromo-2-methyl-4-(tetrahydrofuran-2-yl)quinolone (2378441-97-3)⁴



8-bromo-2-methyl-4-(tetrahydrofuran-2-yl)quinolone
Chemical Formula: $\text{C}_{14}\text{H}_{14}\text{BrNO}$
Exact Mass: 291.0259
Molecular Weight: 292.1760

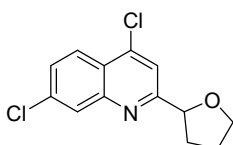
Following the General Procedure A with 8-bromo-2-methylquinoline (44.4 mg, 0.2 mmol), **2k** was obtained as white solid (39.7 mg, 68%), $R_f = 0.5$ (petroleum ether/ethyl acetate = 5:1).

^1H NMR (400 MHz, CDCl_3) δ 7.99 (d, $J = 7.6$ Hz, 1H), 7.80 (d, $J = 8.4$ Hz, 1H), 7.48 (s, 1H), 7.30 (t, $J = 8.4$ Hz, 1H), 5.53 (t, $J = 7.2$ Hz, 1H), 4.20 (dd, $J = 15.2, 8.0$ Hz, 1H), 4.02 (q, $J = 9.6$ Hz, 1H), 2.80 (s, 3H), 2.62 – 2.55 (m, 1H), 2.10 – 2.05 (m, 1H), 2.02 – 1.95 (m, 1H), 1.83 – 1.77

(m, 1H).

^{13}C NMR (101 MHz, CDCl_3) δ 160.4, 149.7, 145.0, 132.7, 125.7, 125.22, 125.18, 122.8, 118.1, 76.7, 69.0, 34.0, 25.9, 26.0.

(2l) 4,7-Dichloro-2-(tetrahydrofuran-2-yl)quinolone (2306793-11-1)¹



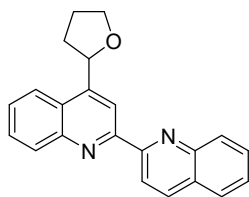
4,7-dichloro-2-(tetrahydrofuran-2-yl)quinolone
Chemical Formula: $\text{C}_{13}\text{H}_{11}\text{Cl}_2\text{NO}$
Exact Mass: 267.0218
Molecular Weight: 268.1370

Following the General Procedure A with 4,7-dichloroquinoline (39.6 mg, 0.2 mmol), **2l** was obtained as colorless oil (33.2 mg, 62%), $R_f = 0.1$ (petroleum ether/ethyl acetate = 4:1).

^1H NMR (400 MHz, CDCl_3) δ 8.12 (d, $J = 9.2$ Hz, 1H), 8.06 (s, 1H), 7.70 (s, 1H), 7.55 – 7.52 (m, 1H), 5.14 – 5.11 (m, 1H), 4.17 – 4.11 (m, 1H), 4.06 – 4.00 (m, 1H), 2.55 – 2.47 (m, 1H), 2.09 – 1.99 (m, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 165.1, 148.5, 143.4, 136.5, 128.1, 128.0, 125.4, 124.0, 118.4, 81.2, 69.3, 33.2, 25.8.

(2m) 4-(Tetrahydrofuran-2-yl)-2,2'-biquinoline



4-(tetrahydrofuran-2-yl)-2,2'-biquinoline
Chemical Formula: C₂₂H₁₈N₂O
Exact Mass: 326.1419
Molecular Weight: 326.3990

Following the General Procedure A with 2,2'-biquinoline (51.3 mg, 0.2 mmol), **2m** was obtained as yellow oil (26.8 mg, 41%), $R_f = 0.6$ (petroleum ether/ethyl acetate = 4:1).

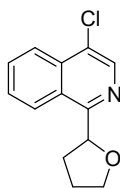
¹H NMR (400 MHz, CDCl₃) δ 8.96 (s, 1H), 8.84 (d, $J = 8.8$ Hz, 1H), 8.33 – 8.28 (m, 3H), 8.0 (d, $J = 8.4$ Hz, 1H), 7.88 (d, $J = 8.0$ Hz, 1H), 7.77 – 7.74 (m, 2H), 7.60 – 7.56 (m, 2H), 5.69 (t, $J = 7.2$ Hz, 1H), 4.39 (q, $J = 14.4$ Hz, 1H), 4.13 (q, $J = 14.8$ Hz, 1H), 2.71 – 2.63 (m, 1H), 2.17 – 2.10 (m, 2H), 2.05 – 1.97 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 156.4, 156.2, 149.8, 148.1, 148.0, 136.6, 130.8, 130.1, 129.4, 129.0, 128.4, 127.6, 126.9, 126.7, 125.9, 123.4, 119.5, 114.9, 77.4, 69.0, 33.8, 26.1.

IR: 2960, 2926, 2853, 1462, 1261, 1094, 1018, 800.

HRMS (ESI) m/z calcd for C₂₂H₁₈N₂O [M+H]⁺: 327.14919, found: 327.14893.

(2n) 4-Chloro-1-(tetrahydrofuran-2-yl)isoquinoline



4-chloro-1-(tetrahydrofuran-2-yl)isoquinoline
Chemical Formula: C₁₃H₁₂ClNO
Exact Mass: 233.0607
Molecular Weight: 233.6950

Following the General Procedure A with 4-chloroisoquinoline (37.2 mg, 0.2 mmol), **2n** was obtained as yellow oil (29.0 mg, 62%), $R_f = 0.5$ (petroleum ether/ethyl acetate = 4:1).

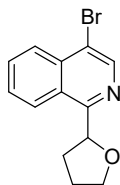
¹H NMR (400 MHz, CDCl₃) δ 8.54 (s, 1H), 8.37 (d, $J = 8.4$ Hz, 1H), 8.23 (d, $J = 8.4$ Hz, 1H), 7.80 (t, $J = 8.0$ Hz, 1H), 7.67 (t, $J = 8.0$ Hz, 1H), 5.68 (t, $J = 7.2$ Hz, 1H), 4.18 – 4.12 (m, 1H), 4.05 – 3.99 (m, 1H), 2.56 – 2.48 (m, 1H), 2.42 – 2.36 (m, 1H), 2.20 – 2.07 (m, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 158.5, 140.1, 133.9, 130.9, 127.9, 127.4, 125.6, 123.9, 78.7, 69.0, 30.6, 26.1.

IR: 3080, 3074, 3045, 2962, 2925, 2856, 1618, 1569, 1257, 1055, 966, 763, 675.

HRMS (ESI) m/z calcd for C₁₃H₁₂ClNO [M+H]⁺: 234.06802, found: 234.06842.

(2o) 4-Bromo-1-(tetrahydrofuran-2-yl)isoquinoline (2408961-61-3)⁵



4-bromo-1-(tetrahydrofuran-2-yl)isoquinoline
Chemical Formula: C₁₃H₁₂BrNO
Exact Mass: 277.0102
Molecular Weight: 278.1490

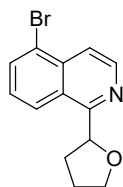
Following the General Procedure A with 4-bromoisoquinoline (41.6 mg, 0.2 mmol), **2o** was obtained as yellow oil (41.2 mg, 74%), $R_f = 0.5$ (petroleum ether/ethyl acetate = 4:1).

¹H NMR (400 MHz, CDCl₃) δ 8.67 (s, 1H), 8.34 (d, $J = 8.4$ Hz, 1H), 8.18 (d, $J = 8.8$ Hz, 1H), 7.77 (t, $J = 7.2$ Hz, 1H), 7.65 (t, $J = 8.0$ Hz, 1H), 5.66 (t, $J = 7.2$ Hz, 1H), 4.17 – 4.11 (m, 1H), 4.04 – 3.99 (m, 1H), 2.56 – 2.47 (m, 1H),

2.42 – 2.33 (m, 1H), 2.20 – 2.04 (m, 2H).

^{13}C NMR (101 MHz, CDCl_3) δ 159.1, 143.0, 135.0, 131.1, 128.0, 127.8, 126.6, 125.6, 119.2, 78.6, 69.0, 30.5, 26.0.

(2p) 5-Bromo-1-(tetrahydrofuran-2-yl)isoquinoline (2095358-00-0)⁵



5-bromo-1-(tetrahydrofuran-2-yl)
isoquinoline

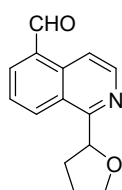
Chemical Formula: $\text{C}_{13}\text{H}_{12}\text{BrNO}$
Exact Mass: 277.0102
Molecular Weight: 278.1490

Following the General Procedure A with 5-bromoisoquinoline (41.6 mg, 0.2 mmol), **2p** was obtained as colorless oil (31.7 mg, 57%), $R_f = 0.4$ (petroleum ether/ethyl acetate = 4:1).

^1H NMR (400 MHz, CDCl_3) δ 8.59 (d, $J = 5.5$ Hz, 1H), 8.34 (d, $J = 8.4$ Hz, 1H), 7.95 (d, $J = 6.4$ Hz, 2H), 7.44 (t, $J = 8.4$ Hz, 1H), 5.70 (t, $J = 7.2$ Hz, 1H), 4.18 – 4.12 (m, 1H), 4.05 – 3.99 (m, 1H), 2.59 – 2.50 (m, 1H), 2.43 – 2.34 (m, 1H), 2.20 – 2.06 (m, 2H).

^{13}C NMR (101 MHz, CDCl_3) δ 160.0, 142.7, 135.6, 133.6, 127.7, 127.4, 125.1, 122.2, 119.4, 79.0, 69.0, 30.6, 26.0.

(2q) 4-(Tetrahydrofuran-2-yl)isoquinoline-5-carbaldehyde



1-(tetrahydrofuran-2-yl)isoquinoline
-5-carbaldehyde

Chemical Formula: $\text{C}_{14}\text{H}_{13}\text{NO}_2$
Exact Mass: 227.0946
Molecular Weight: 227.2630

Following the General Procedure A with isoquinoline-5-carbaldehyde (31.4 mg, 0.2 mmol), **2q** was obtained as colorless oil (11.8 mg, 26%), $R_f = 0.2$ (petroleum ether/ethyl acetate = 4:1).

^1H NMR (400 MHz, CDCl_3) δ 10.39 (s, 1H), 8.97 (d, $J = 6.0$ Hz, 1H), 8.72 – 8.66 (m, 2H), 8.19 (d, $J = 7.2$ Hz, 1H), 7.78 (t, $J = 8.0$ Hz, 1H), 5.71 (t, $J = 7.2$ Hz, 1H), 4.18 – 4.12 (m, 1H), 4.06 – 4.00 (m, 1H), 2.67 – 2.58 (m, 1H), 2.42 – 2.36 (m, 1H), 2.23 – 2.09 (m, 2H).

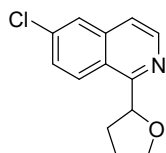
^{13}C NMR (101 MHz, CDCl_3) δ 192.6, 159.8, 144.6, 139.1, 134.2, 132.5, 130.8, 126.8, 126.1, 117.2, 79.4, 69.0, 30.2, 26.1.

Melting point ($^\circ\text{C}$): 155.6 – 108.0 $^\circ\text{C}$.

IR: 2989, 2987, 2964, 2879, 2750, 1678, 1564, 1225, 1180, 1057, 852, 769, 660.

HRMS (ESI) m/z calcd for $\text{C}_{14}\text{H}_{13}\text{N}_2\text{O}$ $[\text{M}+\text{H}]^+$: 228.10191, found: 228.10199.

(2r) 6-Chloro-1-(tetrahydrofuran-2-yl)isoquinoline (2095357-99-4)⁵



6-chloro-1-(tetrahydrofuran-2-yl)
isoquinoline

Chemical Formula: $\text{C}_{13}\text{H}_{12}\text{ClNO}$
Exact Mass: 233.0607
Molecular Weight: 233.6950

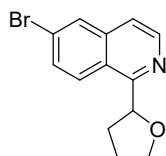
Following the General Procedure A with 6-chloroisoquinoline (32.7 mg, 0.2 mmol), **2r** was obtained as yellow oil (15.0 mg, 32%), $R_f = 0.4$ (petroleum ether/ethyl acetate = 4:1).

^1H NMR (400 MHz, CDCl_3) δ 8.50 (d, $J = 5.6$ Hz, 1H), 8.32 (d, $J = 8.8$ Hz, 1H), 7.81 (s, 1H), 7.54 (d, $J = 9.2$ Hz, 1H), 7.50 (d, $J = 5.6$ Hz, 1H), 5.65 (t, $J = 6.8$ Hz, 1H), 4.19

– 4.13 (m, 1H), 4.05 – 4.00 (m, 1H), 2.57 – 2.48 (m, 1H), 2.43 – 2.35 (m, 1H), 2.22 – 2.07 (m, 2H).

^{13}C NMR (101 MHz, CDCl_3) δ 159.7, 142.4, 137.4, 136.2, 128.1, 127.3, 126.0, 124.8, 119.7, 79.2, 69.0, 30.6, 26.1.

(2s) 6-Bromo-1-(tetrahydrofuran-2-yl)isoquinoline (2095357-98-3)⁶



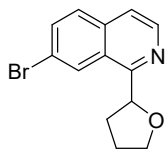
6-bromo-1-(tetrahydrofuran-2-yl)
isoquinoline
Chemical Formula: $\text{C}_{13}\text{H}_{12}\text{BrNO}$
Exact Mass: 277.0102
Molecular Weight: 278.1490

Following the General Procedure A with 6-bromoisoquinoline (41.6 mg, 0.2 mmol), **2s** was obtained as white solid (39.5 mg, 71%), $R_f = 0.2$ (petroleum ether/ethyl acetate = 4:1).

^1H NMR (400 MHz, CDCl_3) δ 8.49 (d, $J = 5.6$ Hz, 1H), 8.23 (d, $J = 9.2$ Hz, 1H), 7.97 (d, $J = 1.2$ Hz, 1H), 7.67 – 7.64 (m, 1H), 7.47 (d, $J = 6.0$ Hz, 1H), 5.63 (t, $J = 7.2$ Hz, 1H), 4.17 – 4.11 (m, 1H), 4.04 – 3.98 (m, 1H), 2.56 – 2.48 (m, 1H), 2.41 – 2.33 (m, 1H), 2.19 – 2.06 (m, 2H).

^{13}C NMR (101 MHz, CDCl_3) δ 159.8, 142.4, 137.7, 130.6, 129.3, 127.2, 125.0, 124.6, 119.4, 79.1, 69.0, 30.5, 26.0.

(2t) 7-Bromo-1-(tetrahydrofuran-2-yl)isoquinoline (2095358-02-2)⁶



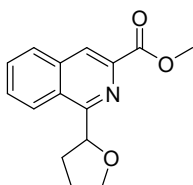
7-bromo-1-(tetrahydrofuran-2-yl)
isoquinoline
Chemical Formula: $\text{C}_{13}\text{H}_{12}\text{BrNO}$
Exact Mass: 277.0102
Molecular Weight: 278.1490

Following the General Procedure A with 7-bromoisoquinoline (41.6 mg, 0.2 mmol), **2t** was obtained as white solid (31.2 mg, 56%), $R_f = 0.3$ (petroleum ether/ethyl acetate = 4:1).

^1H NMR (400 MHz, CDCl_3) δ 8.53 – 8.50 (m, 2H), 7.75 – 7.68 (m, 2H), 7.54 (d, $J = 6.0$ Hz, 1H), 5.60 (t, $J = 7.2$ Hz, 1H), 4.18 – 4.13 (m, 1H), 4.06 – 4.00 (m, 1H), 2.58 – 2.50 (m, 1H), 2.42 – 2.34 (m, 1H), 2.22 – 2.07 (m, 2H).

^{13}C NMR (100 MHz, CDCl_3) δ 158.6, 141.7, 134.9, 133.3, 128.8, 127.8, 127.5, 120.9, 120.1, 79.0, 68.9, 30.4, 26.0.

(2u) Methyl 1-(tetrahydrofuran-2-yl)isoquinoline-3-carboxylate (2095358-05-5)⁶



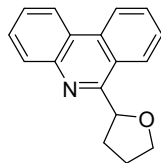
methyl 1-(tetrahydrofuran-2-yl)
isoquinoline-3-carboxylate
Chemical Formula: $\text{C}_{15}\text{H}_{15}\text{NO}_3$
Exact Mass: 257.1052
Molecular Weight: 257.2890

Following the General Procedure A with methyl isoquinoline-3-carboxylate (37.4 mg, 0.2 mmol), **2u** was obtained as white solid (27.3 mg, 53%), $R_f = 0.3$ (petroleum ether/ethyl acetate = 4:1).

^1H NMR (400 MHz, CDCl_3) δ 8.52 – 8.50 (m, 2H), 7.98 – 7.95 (m, 1H), 7.77 – 7.71 (m, 2H), 5.68 (t, $J = 7.2$ Hz, 1H), 4.17 (dd, $J = 14.8, 7.6$ Hz, 1H), 4.06 – 4.00 (m, 4H), 2.74 – 2.65 (m, 1H), 2.47 – 2.38 (m, 1H), 2.27 – 2.08 (m, 2H).

^{13}C NMR (101 MHz, CDCl_3) δ 166.4, 160.0, 139.9, 136.4, 130.6, 129.3, 128.8, 128.1, 126.1, 124.2, 80.4, 69.0, 52.7, 30.3, 26.1.

(2v) 6-(Tetrahydrofuran-2-yl)phenanthridine (1588454-61-8)⁵



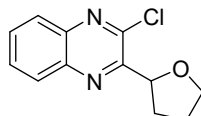
6-(tetrahydrofuran-2-yl)
phenanthridine
Chemical Formula: C₁₇H₁₅NO
Exact Mass: 249.1154
Molecular Weight: 249.3130

Following the General Procedure A with phenanthridine (35.8 mg, 0.2 mmol), **2v** was obtained as colorless oil (30.4 mg, 61%), *R_f* = 0.6 (petroleum ether/ethyl acetate = 4:1).

¹H NMR (400 MHz, CDCl₃) δ 8.63 (d, *J* = 8.4 Hz, 1H), 8.54 (d, *J* = 8.0 Hz, 1H), 8.45 (d, *J* = 8.4 Hz, 1H), 8.21 (d, *J* = 8.0 Hz, 1H), 7.82 (t, *J* = 7.2 Hz, 1H), 7.74 – 7.62 (m, 3H), 5.78 (t, *J* = 6.8 Hz, 1H), 4.24 – 4.19 (m, 1H), 4.10 – 4.05 (m, 1H), 2.79 – 2.71 (m, 1H), 2.47 – 2.38 (m, 1H), 2.23 – 2.18 (m, 1H), 2.16 – 2.10 (m, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 159.2, 143.1, 133.2, 130.32, 130.26, 128.4, 127.1, 126.8, 126.4, 124.7, 124.0, 122.3, 121.8, 79.5, 69.0, 30.0, 25.9.

(2w) 2-Chloro-3-(tetrahydrofuran-2-yl)quinoxaline (2770705-43-4)⁵



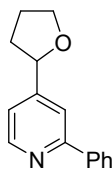
Chemical Formula: C₁₂H₁₁ClN₂O
Exact Mass: 234.0560
Molecular Weight: 234.6830

Following the General Procedure A with 2-chloroquinoxaline (32.9 mg, 0.2 mmol), **2w** was obtained as colorless oil (21.6 mg, 46%), *R_f* = 0.2 (petroleum ether/ethyl acetate = 6:1).

¹H NMR (400 MHz, CDCl₃) δ 8.15 – 8.13 (m, 1H), 8.00 – 7.98 (m, 1H), 7.76 – 7.73 (m, 2H), 5.55 (t, *J* = 6.0 Hz, 1H), 4.29 – 4.23 (m, 1H), 4.08 – 4.03 (m, 1H), 2.53 – 2.48 (m, 1H), 2.27 – 2.22 (m, 1H), 2.18 – 2.06 (m, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 154.5, 146.0, 141.3, 140.6, 130.6, 130.1, 129.3, 128.0, 78.0, 69.4, 30.9, 25.7.

(2x) 2-Phenyl-4-(tetrahydrofuran-2-yl)pyridine (1795742-55-0)⁷



Chemical Formula: C₁₅H₁₅NO
Exact Mass: 225.1154
Molecular Weight: 225.2910

Following the General Procedure A with 2-phenylpyridine (31.0 mg, 0.2 mmol), **2x** was obtained as yellow oil (18.5 mg, 41%), *R_f* = 0.3 (petroleum ether/ethyl acetate = 4:1).

¹H NMR (400 MHz, CDCl₃): δ 8.63 (d, *J* = 5.2 Hz, 1H), 8.00 (d, *J* = 6.8 Hz, 2H), 7.70 (s, 1H), 7.49 – 7.39 (m, 3H), 7.19 (d, *J* = 4.4 Hz, 1H), 4.97 (t, *J* = 7.2 Hz, 1H), 4.15 – 4.10 (m, 1H), 4.02–3.96 (m, 1H), 2.44 – 2.38 (m, 1H), 2.06–1.98 (m, 2H), 1.84–1.79 (m, 1H).

¹³C NMR (101 MHz, CDCl₃): δ 157.5, 153.7, 149.5, 139.3, 128.93, 128.9, 127.0, 119.0, 117.3, 79.2, 69.0, 34.3, 25.8.

5 References

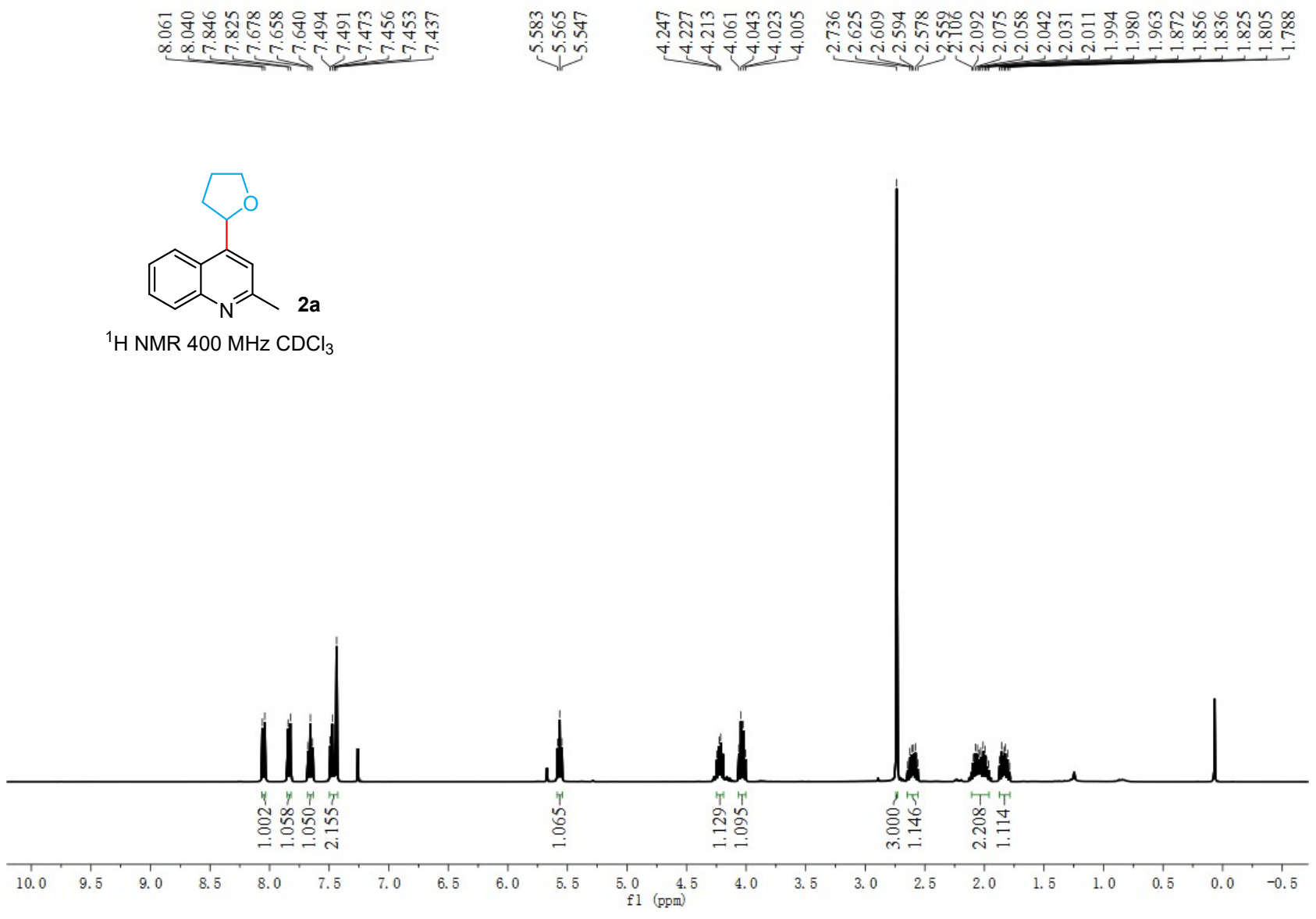
1. J. Zhou, Y. Zou, P. Zhou, Z. Chen and J. Li, *Org. Chem. Front.*, 2019, **6**, 1594.
2. C. Y. Huang, J. Li, W. Liu and C. J. Li, *Chem. Sci.*, 2019, **10**, 5018–5024.
3. X. Li, C. Liu, S. Guo, W. Wang and Y. Zhang, *Eur. J. Org. Chem.*, 2021, 411–421.
4. S. Wang, Y. Fan, H. Zhao, J. Wang, S. Zhang and W. Wang, *Synlett*, 2019, **30**, 2096–2100.
5. L. Li, X. Song, M. F. Qi and B. Sun, *Tetrahedron.*, 2022, **99**, 153846.
6. S. Liu, A. Liu, Y. Zhang and W. Wang, *Chem. Sci.*, 2017, **8**, 4044.
7. Z. Zhou, Y. Wu, P. Yang, S. Deng, Q. Zhang and D. Li, *ChemistrySelect*, 2021, **6**, 2770–2773.

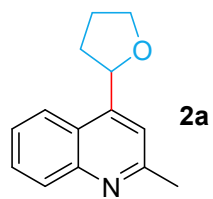
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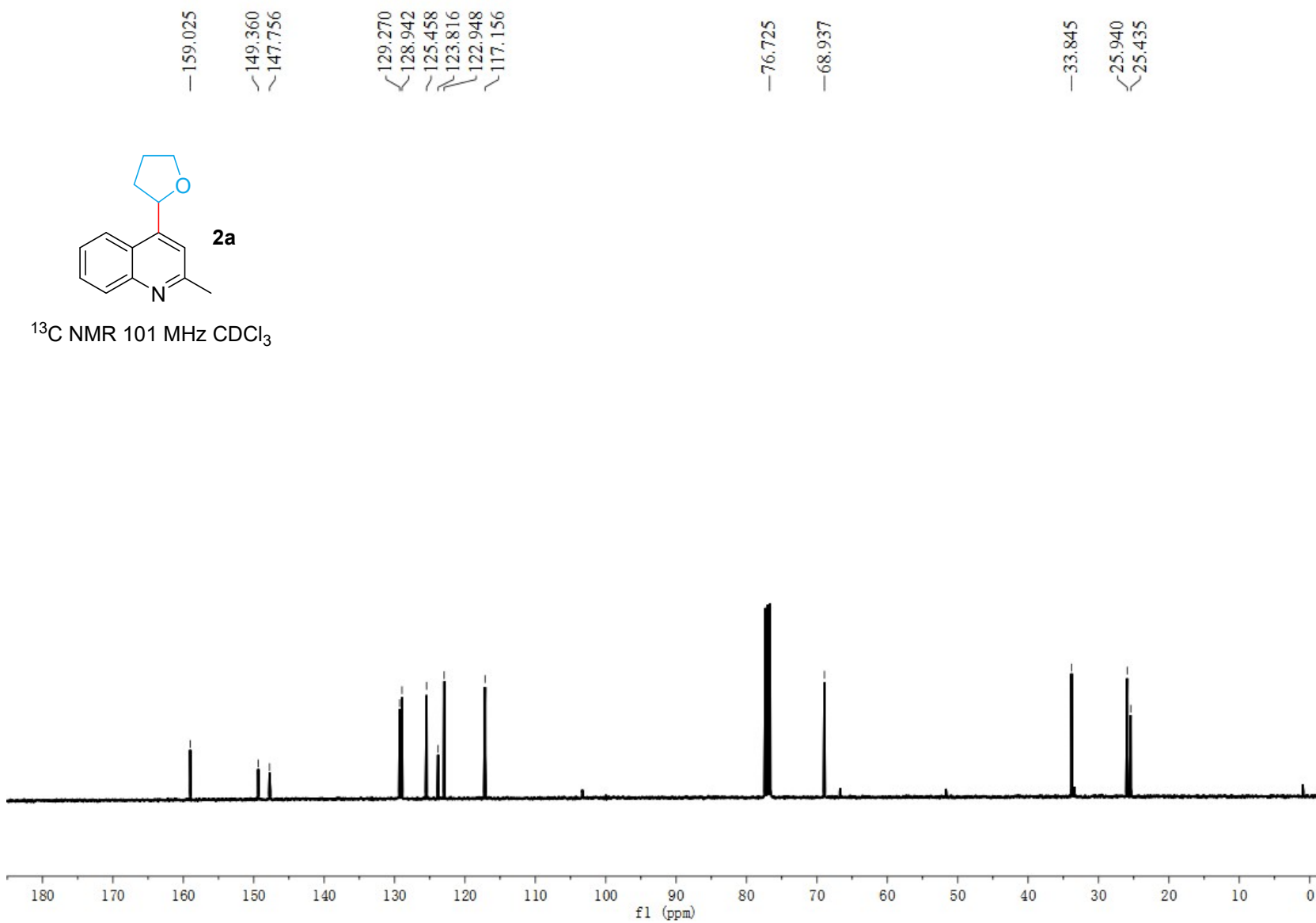
NMR

spectra





^{13}C NMR 101 MHz CDCl_3

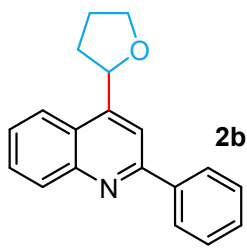


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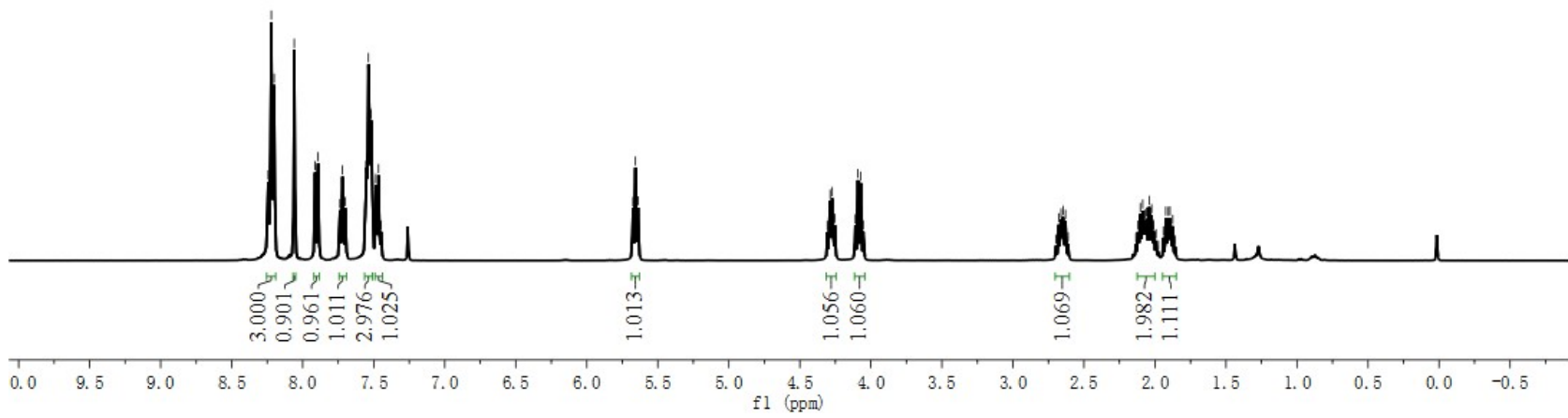
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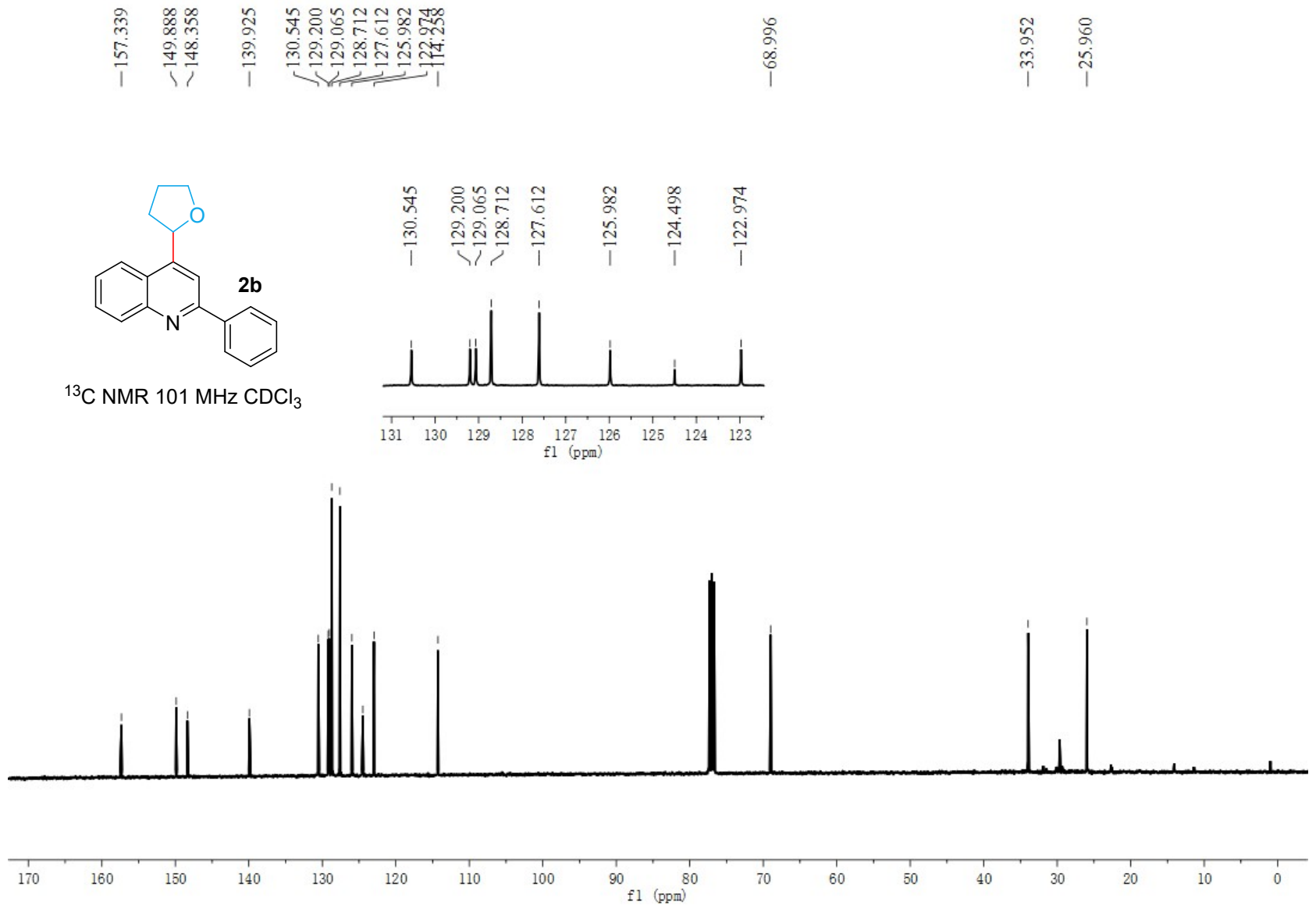
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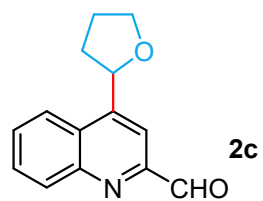
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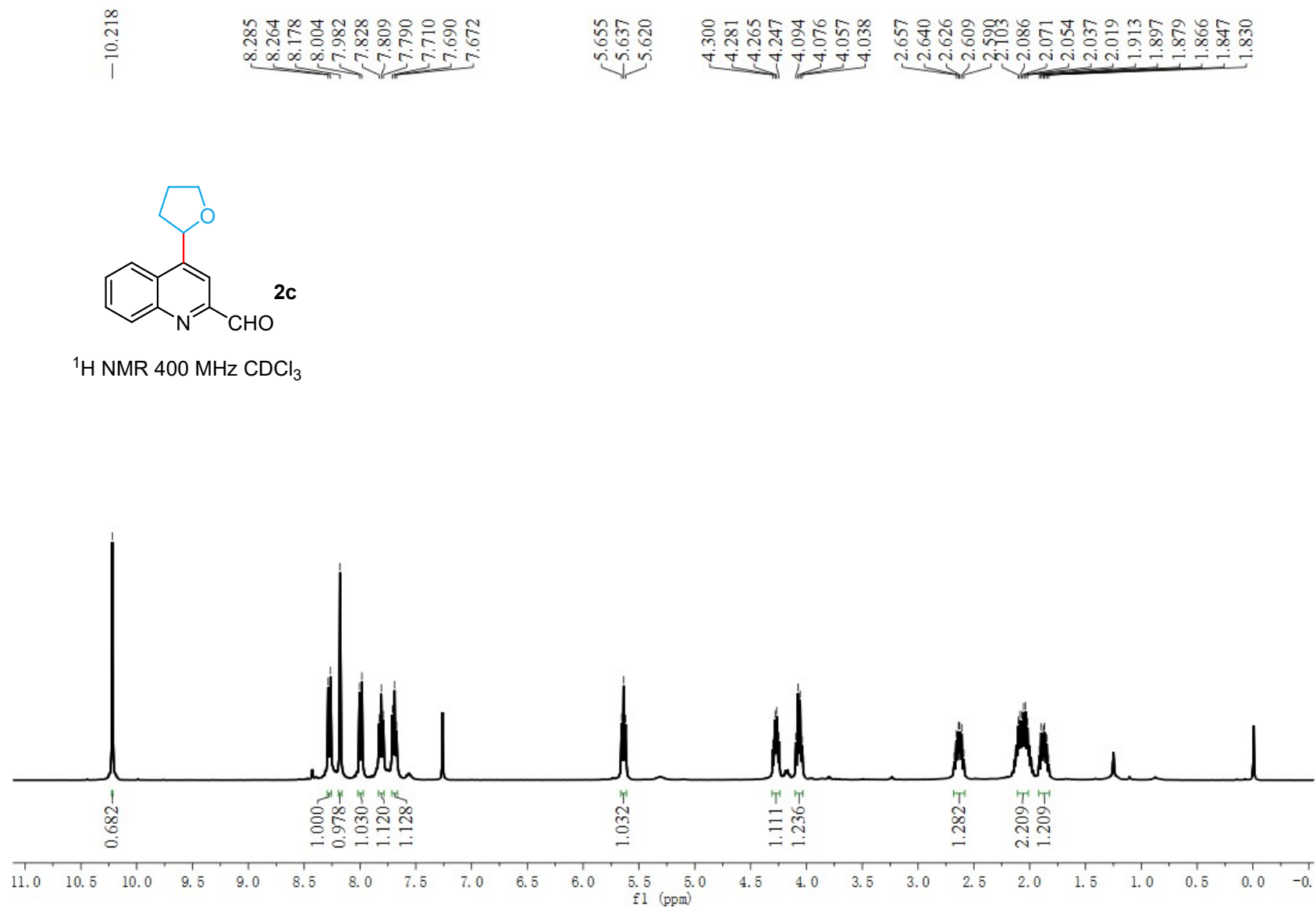
¹H NMR 400 MHz CDCl₃

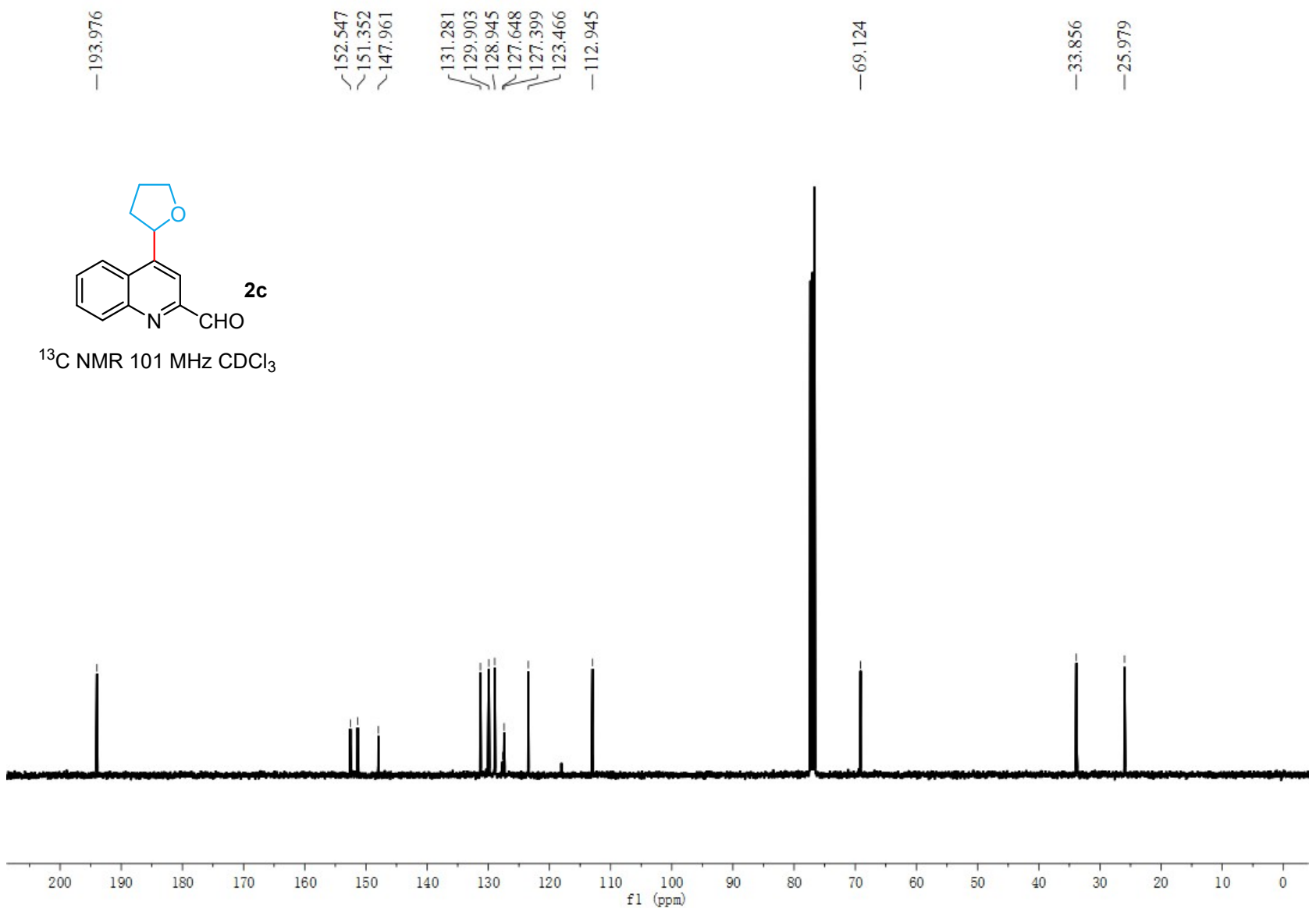
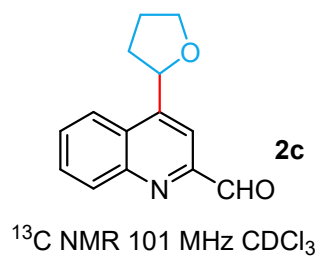


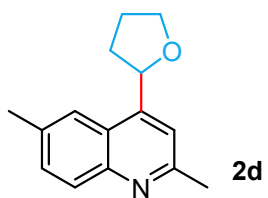




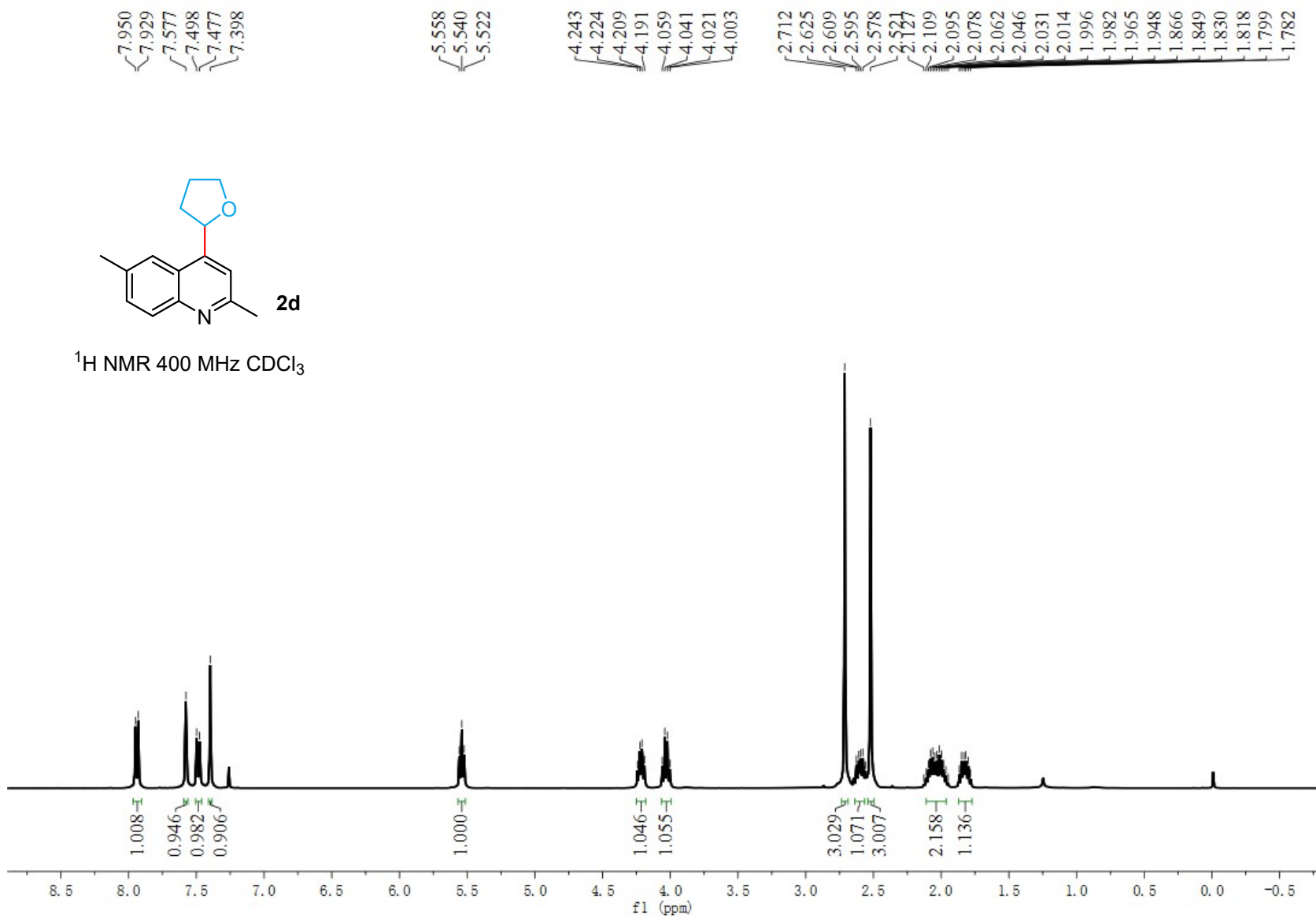
$^1\text{H NMR}$ 400 MHz CDCl_3

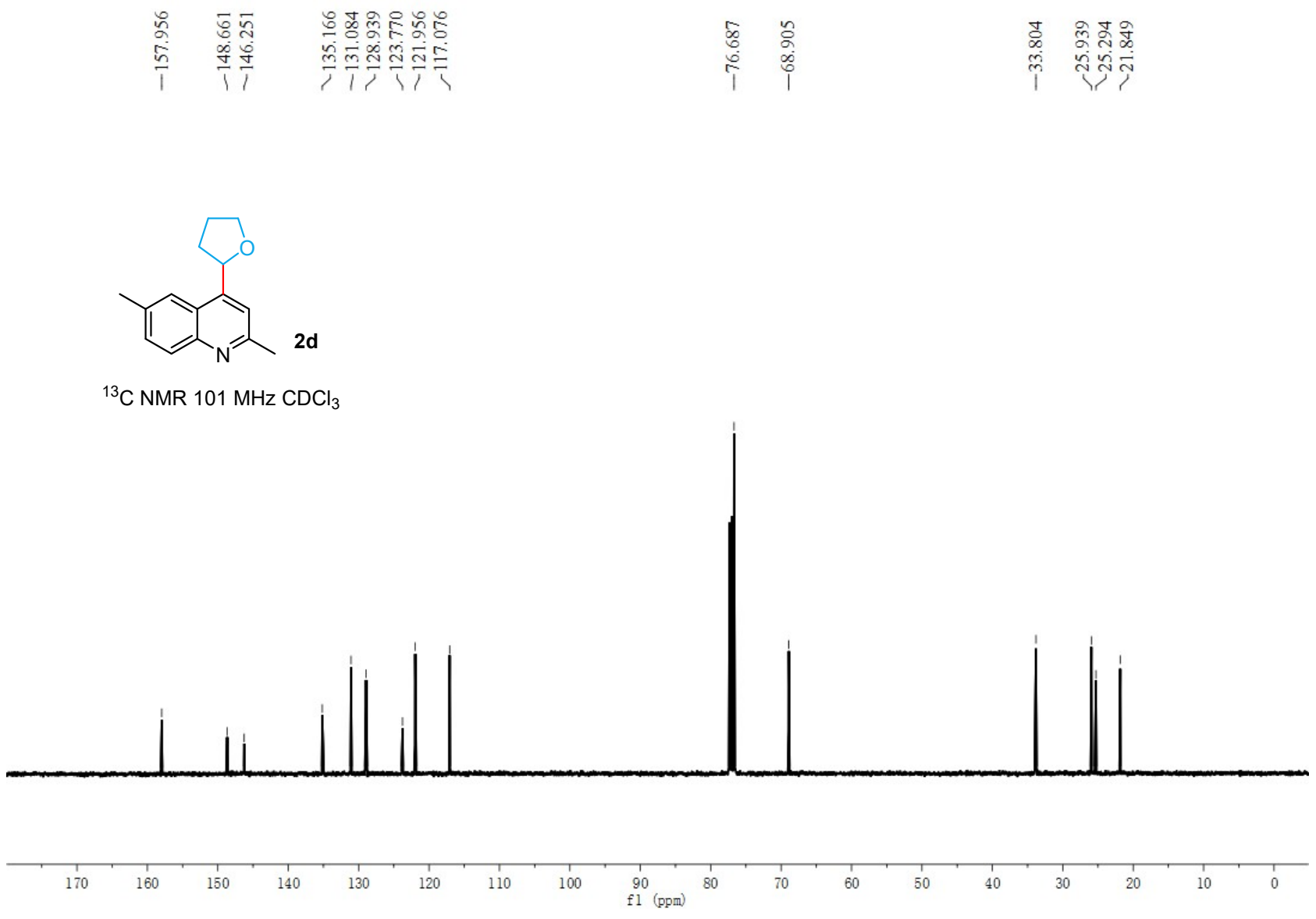
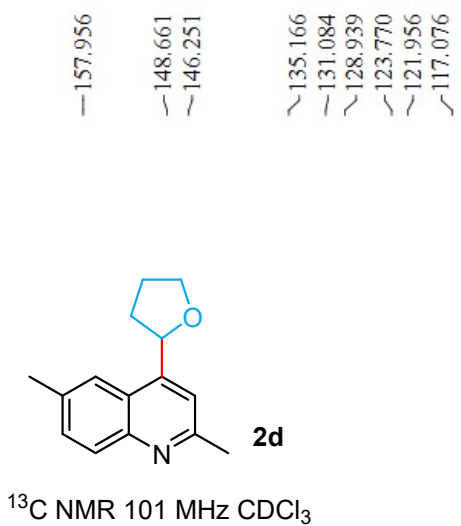


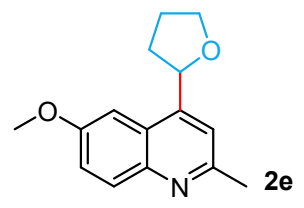




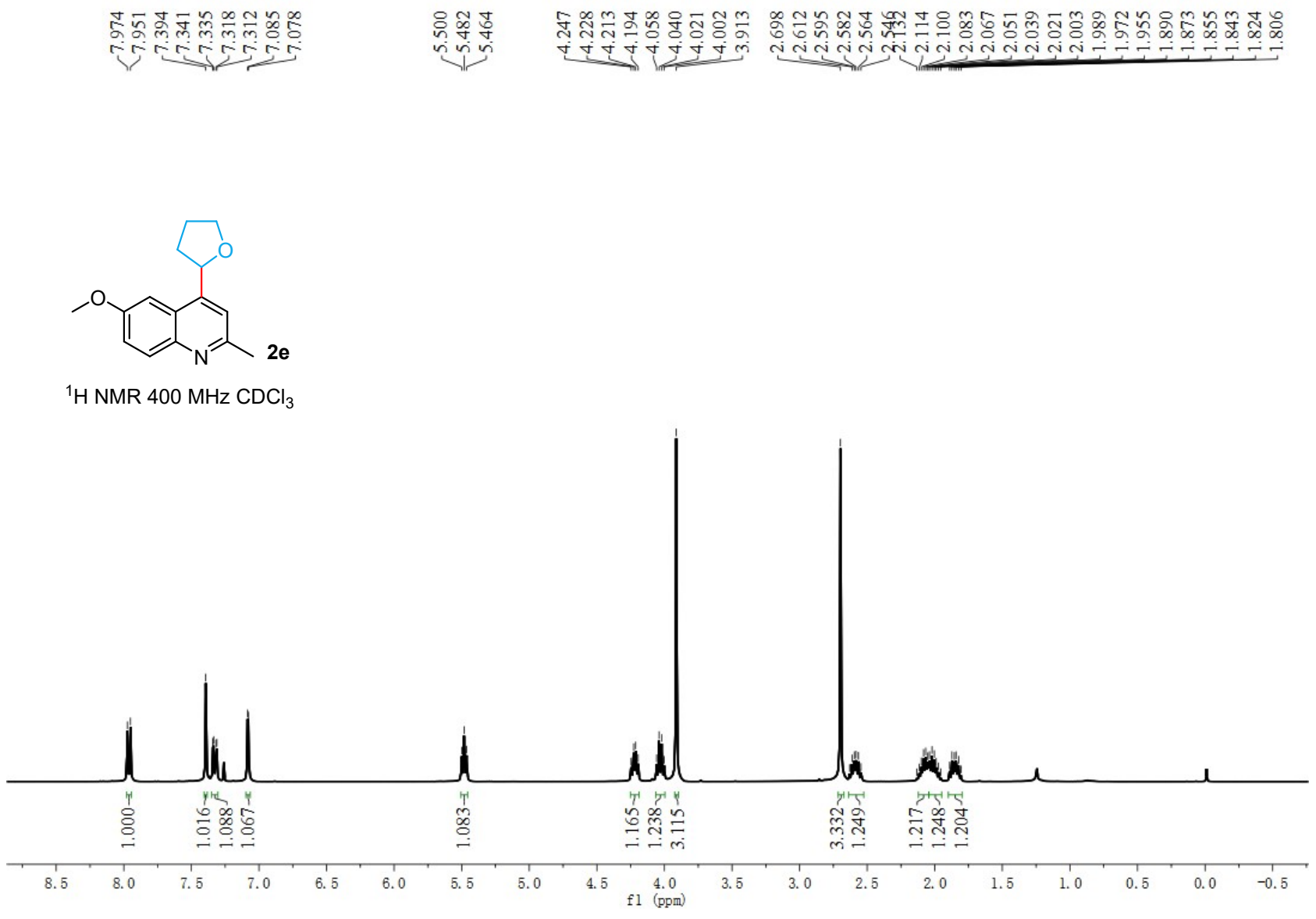
$^1\text{H NMR}$ 400 MHz CDCl_3

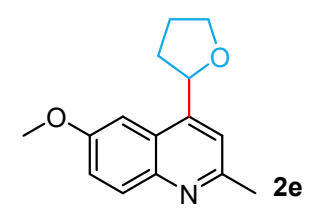




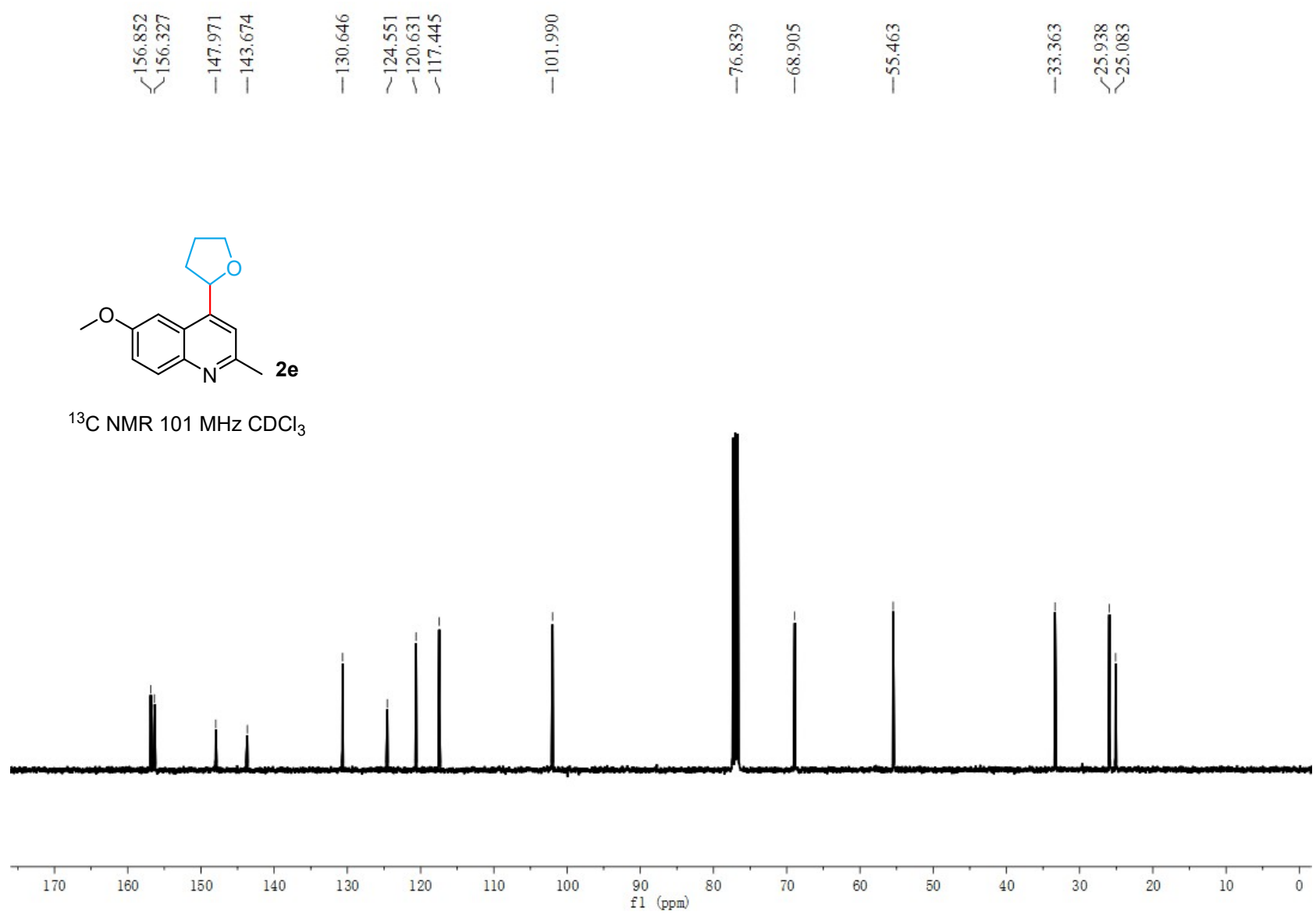


¹H NMR 400 MHz CDCl₃





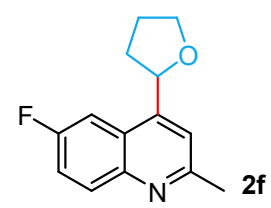
¹³C NMR 101 MHz CDCl₃



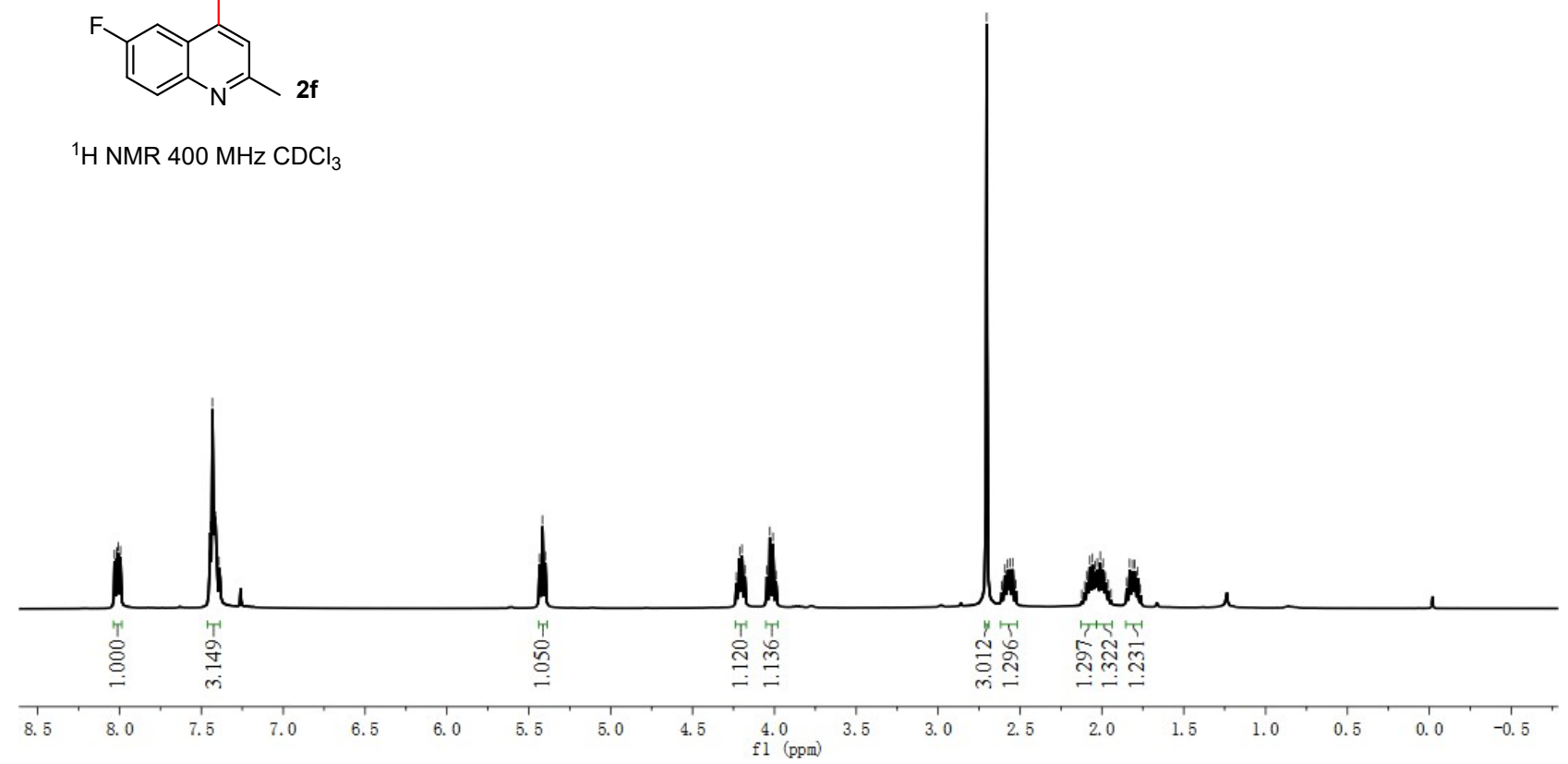
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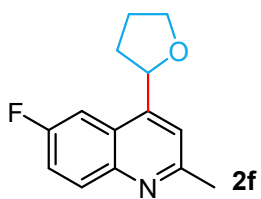
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1.763

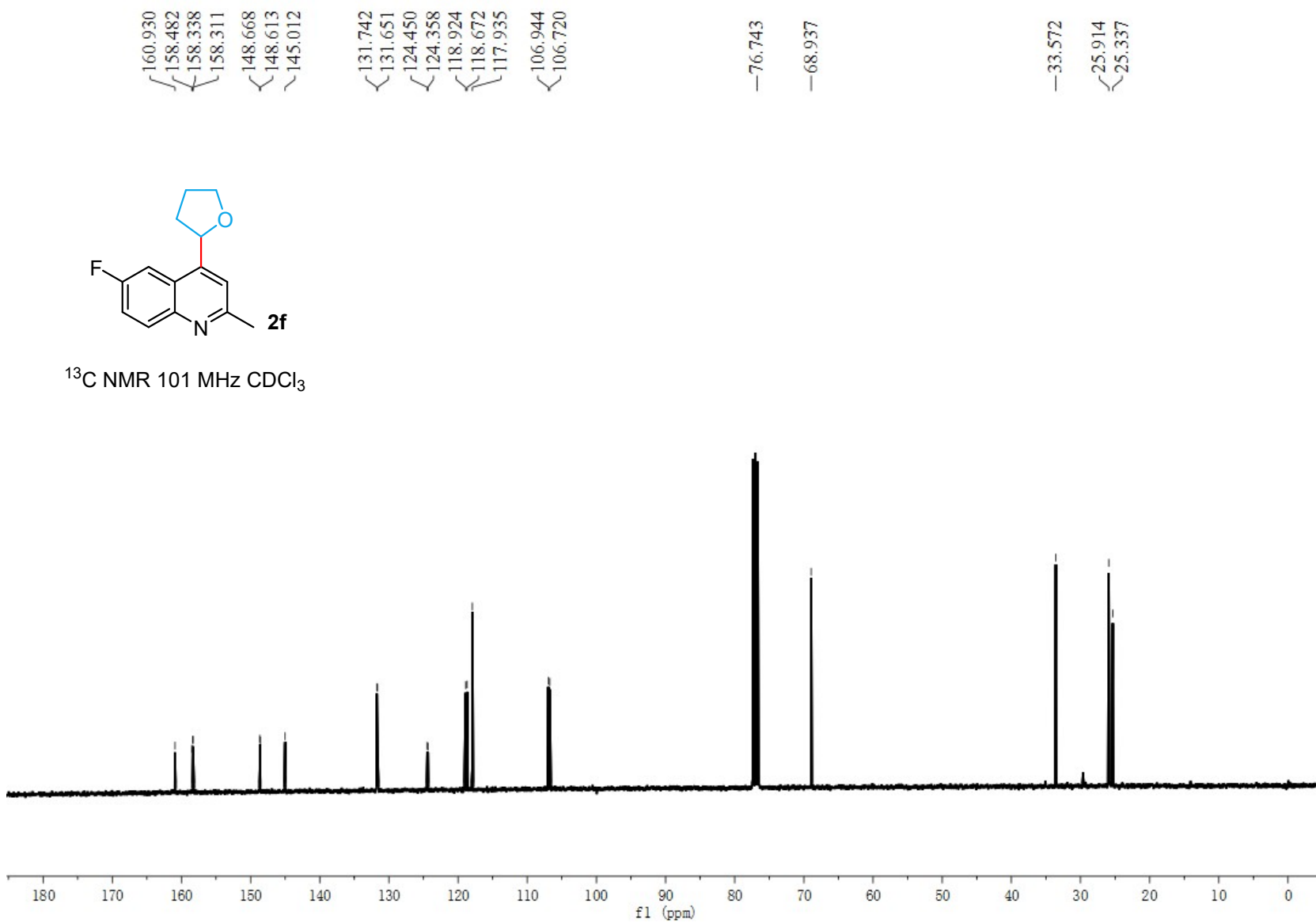


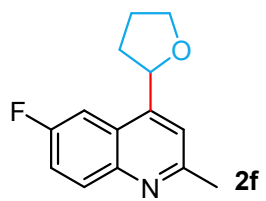
¹H NMR 400 MHz CDCl₃



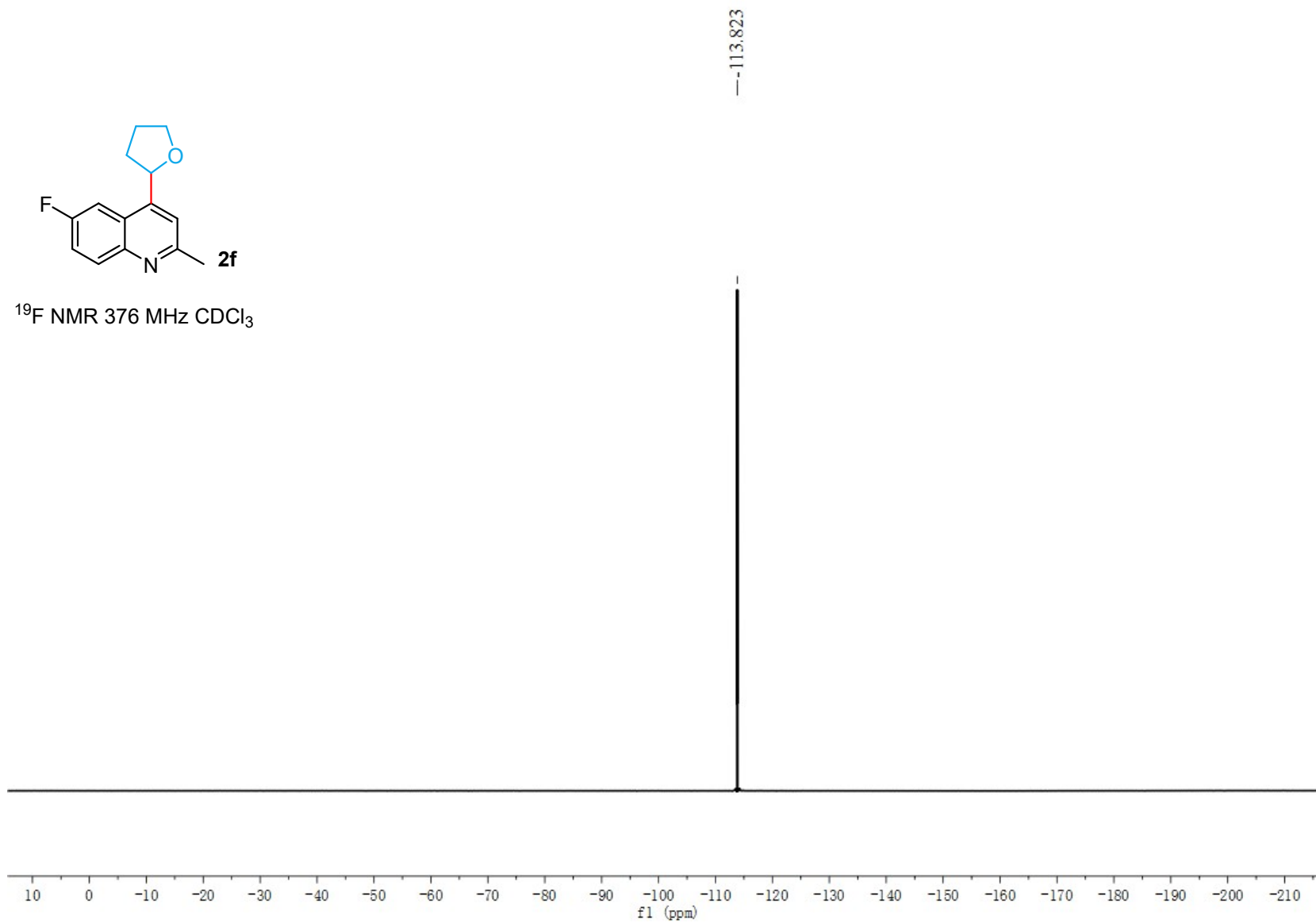


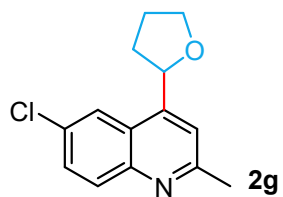
^{13}C NMR 101 MHz CDCl_3



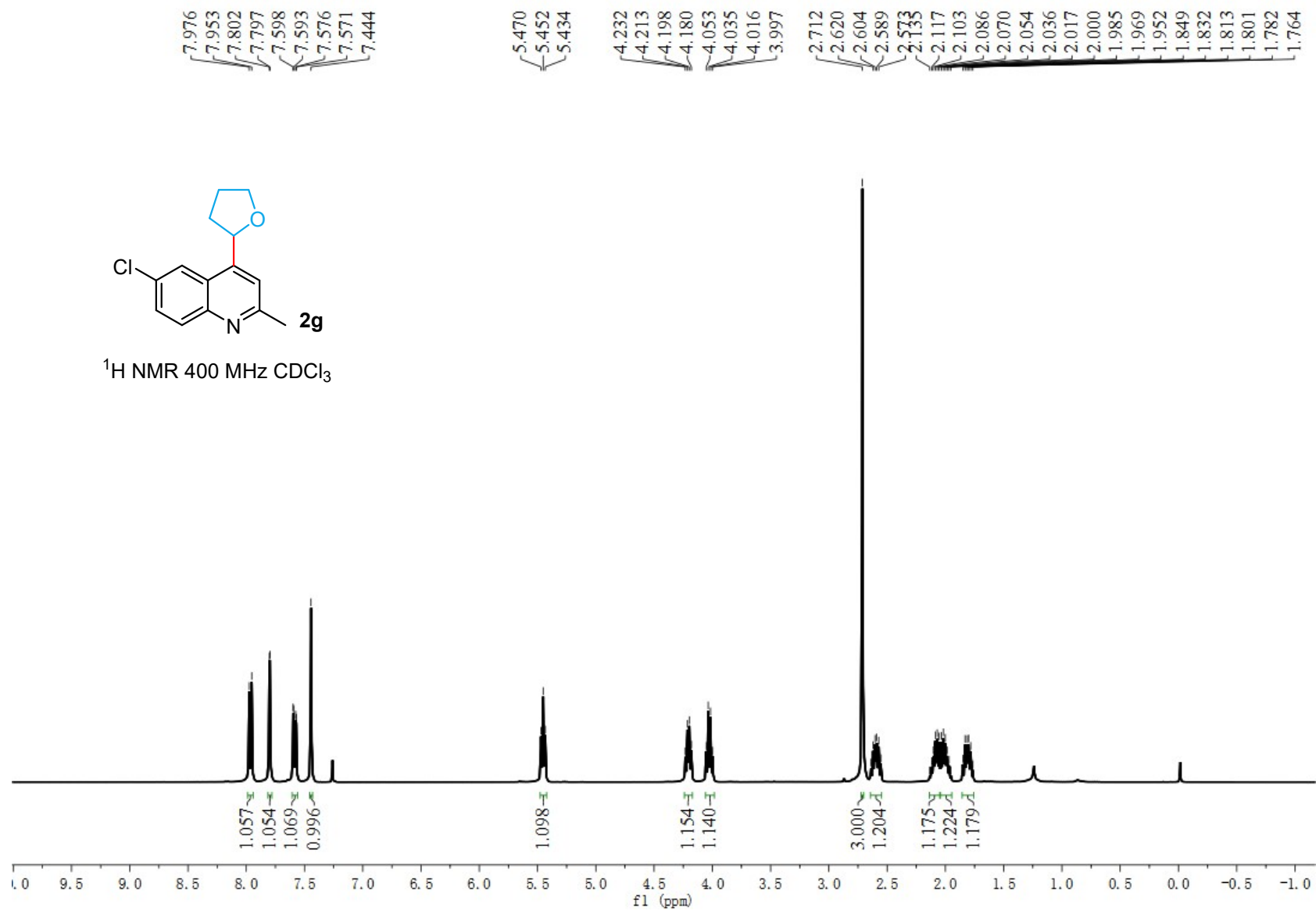


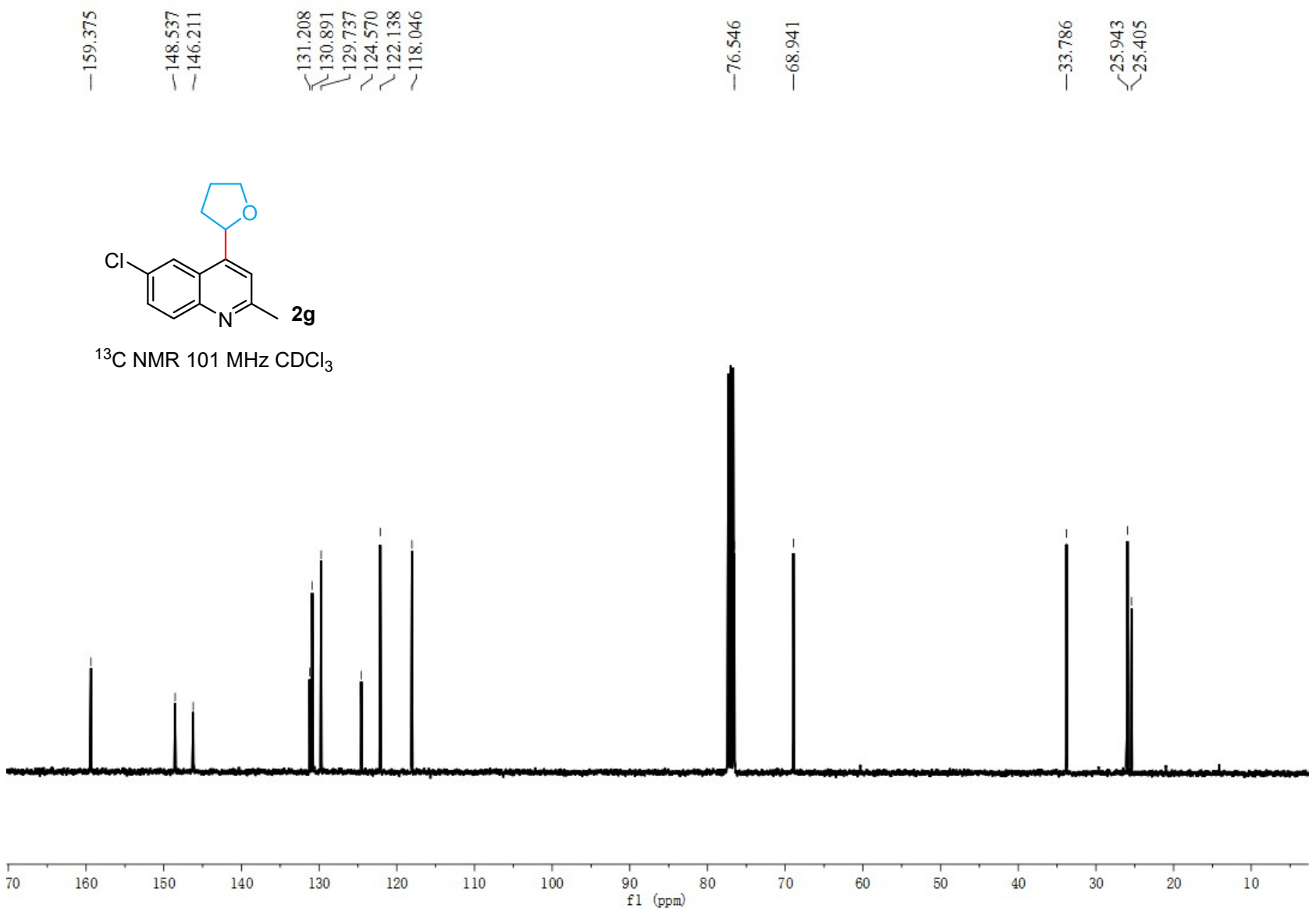
^{19}F NMR 376 MHz CDCl_3

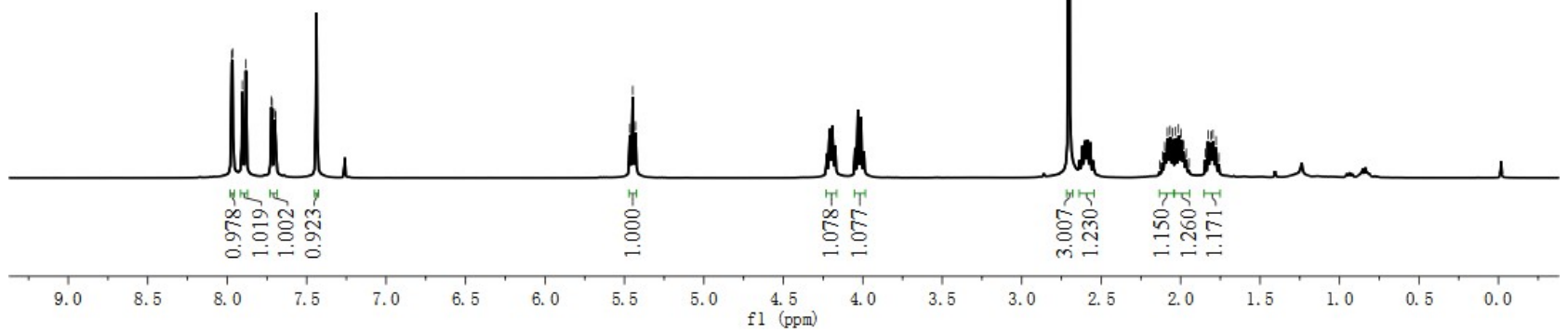
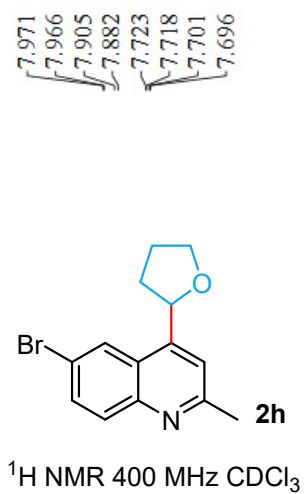


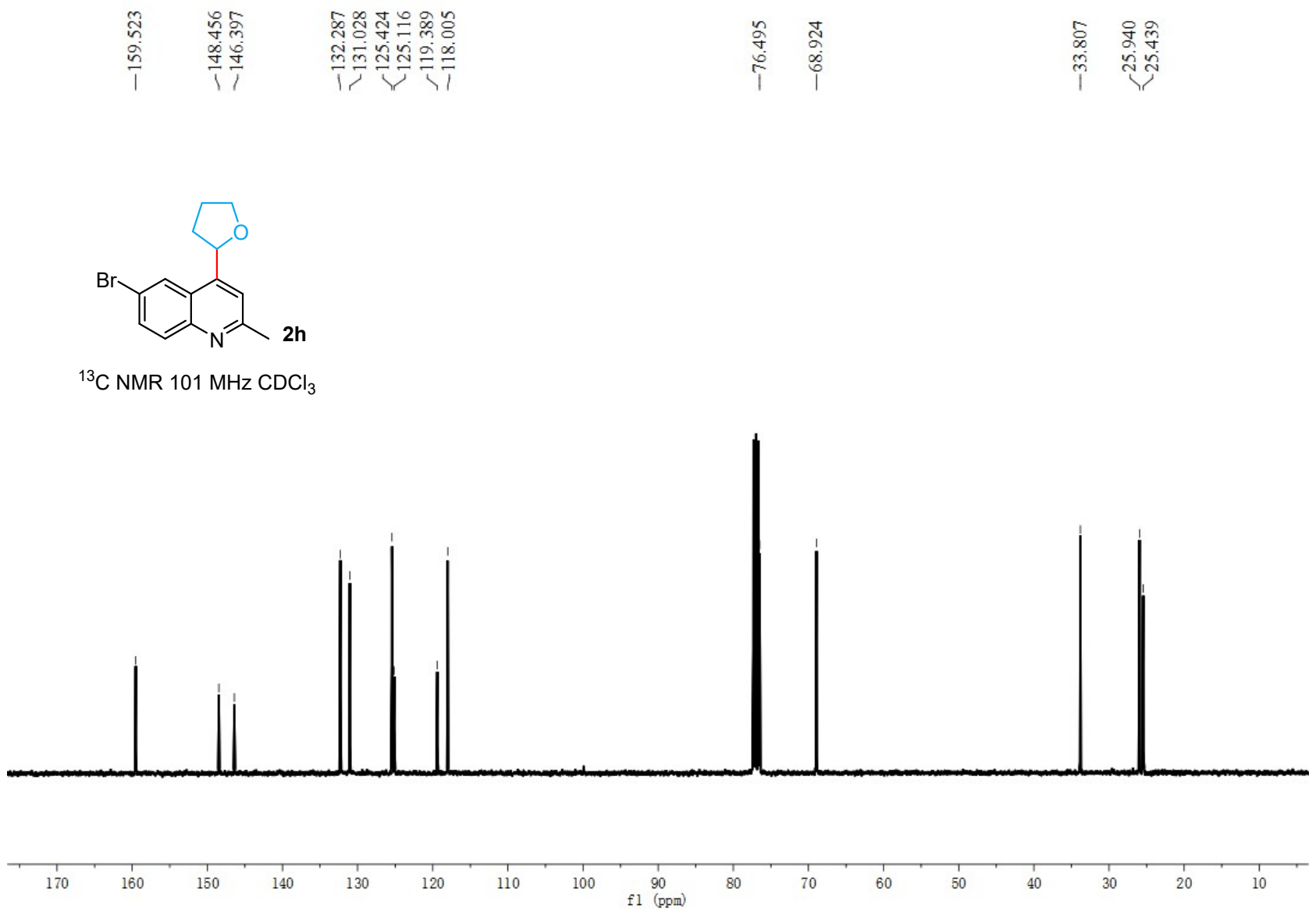
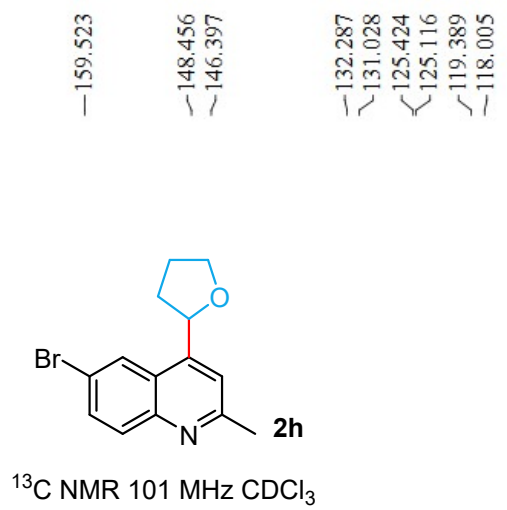


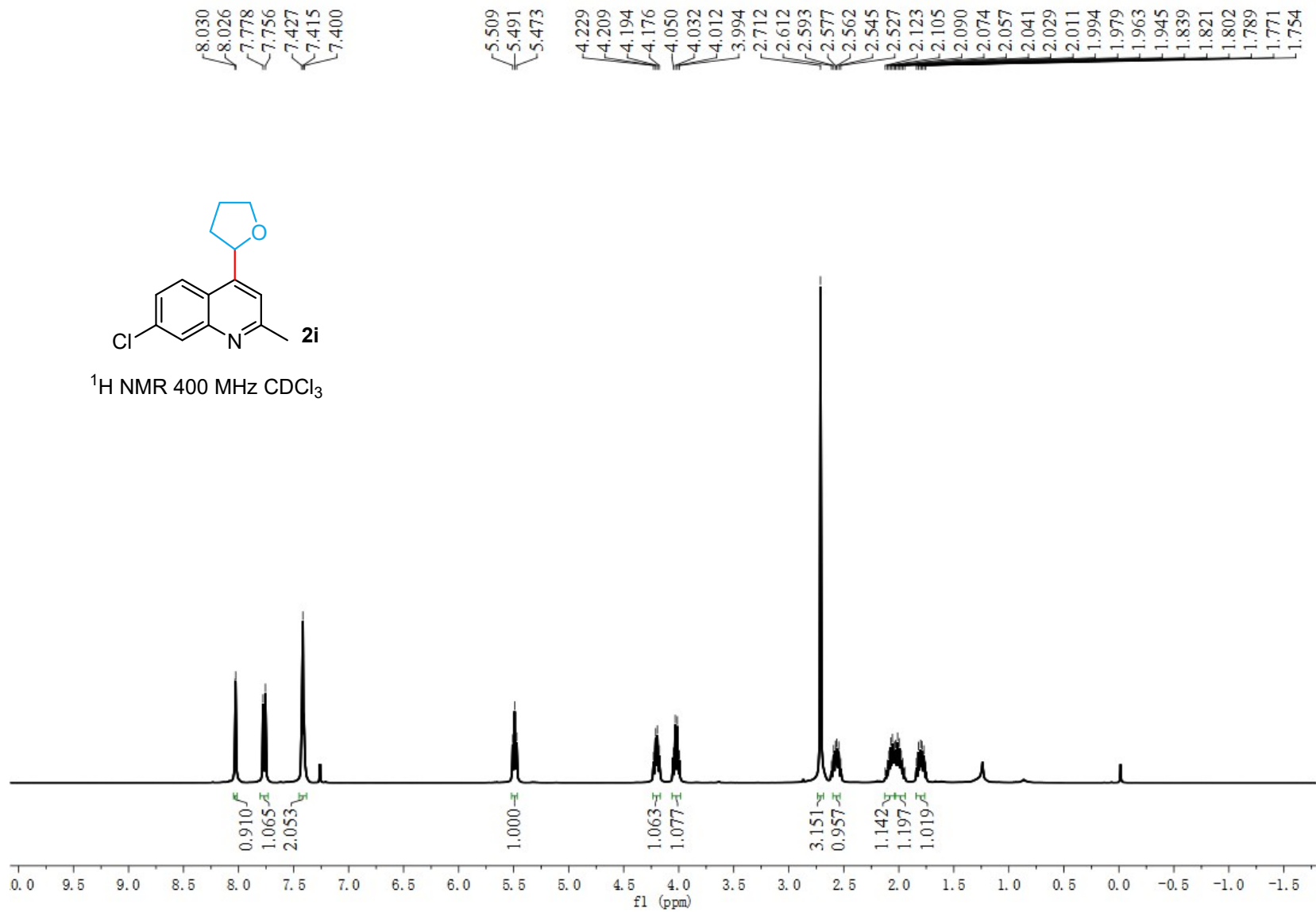
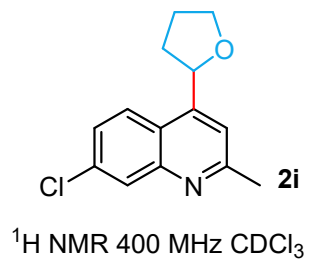
$^1\text{H NMR}$ 400 MHz CDCl_3







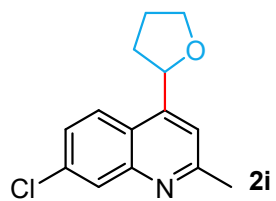




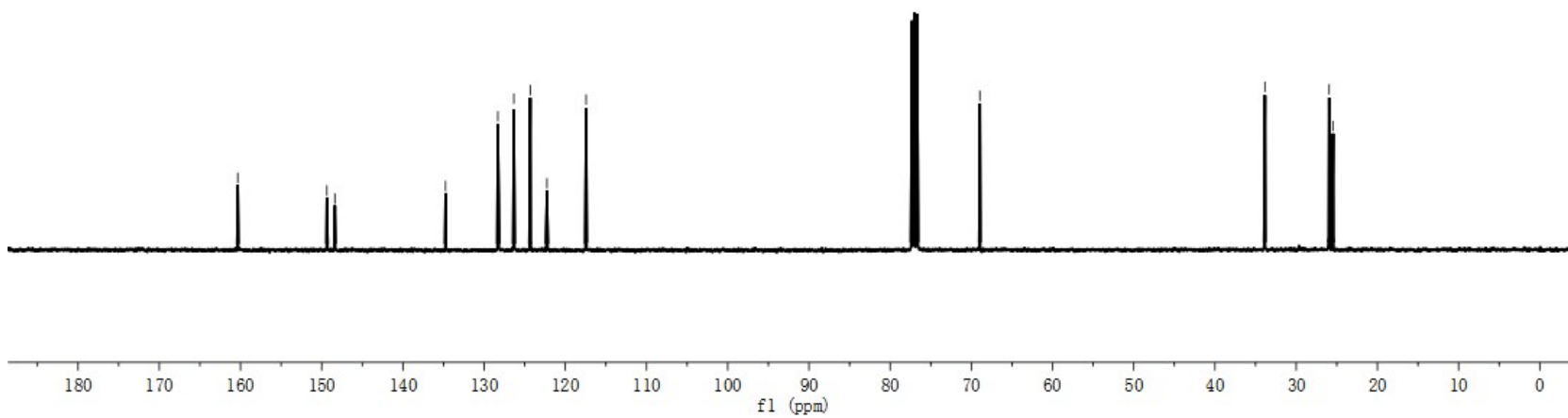
—160.334
—149.362
—148.381
—134.750
—128.309
—126.341
—124.324
—122.256
—117.441

—76.612
—68.952

—33.845
—25.936
—25.454



¹³C NMR 101 MHz CDCl₃

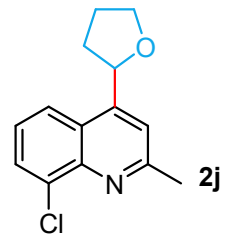


7.795
7.785
7.777
7.764
7.510
7.407
7.387
7.367

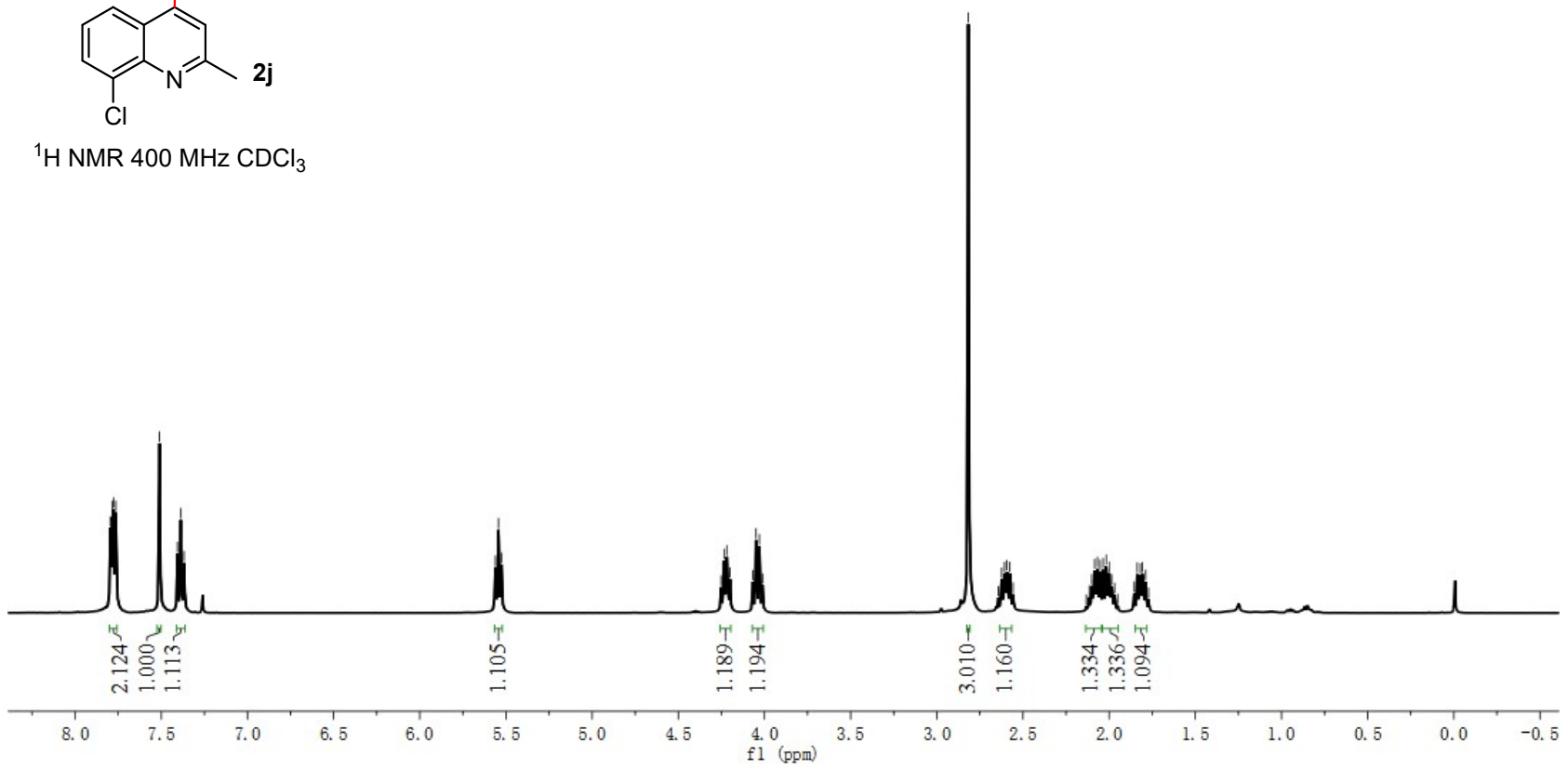
5.562
5.544
5.526

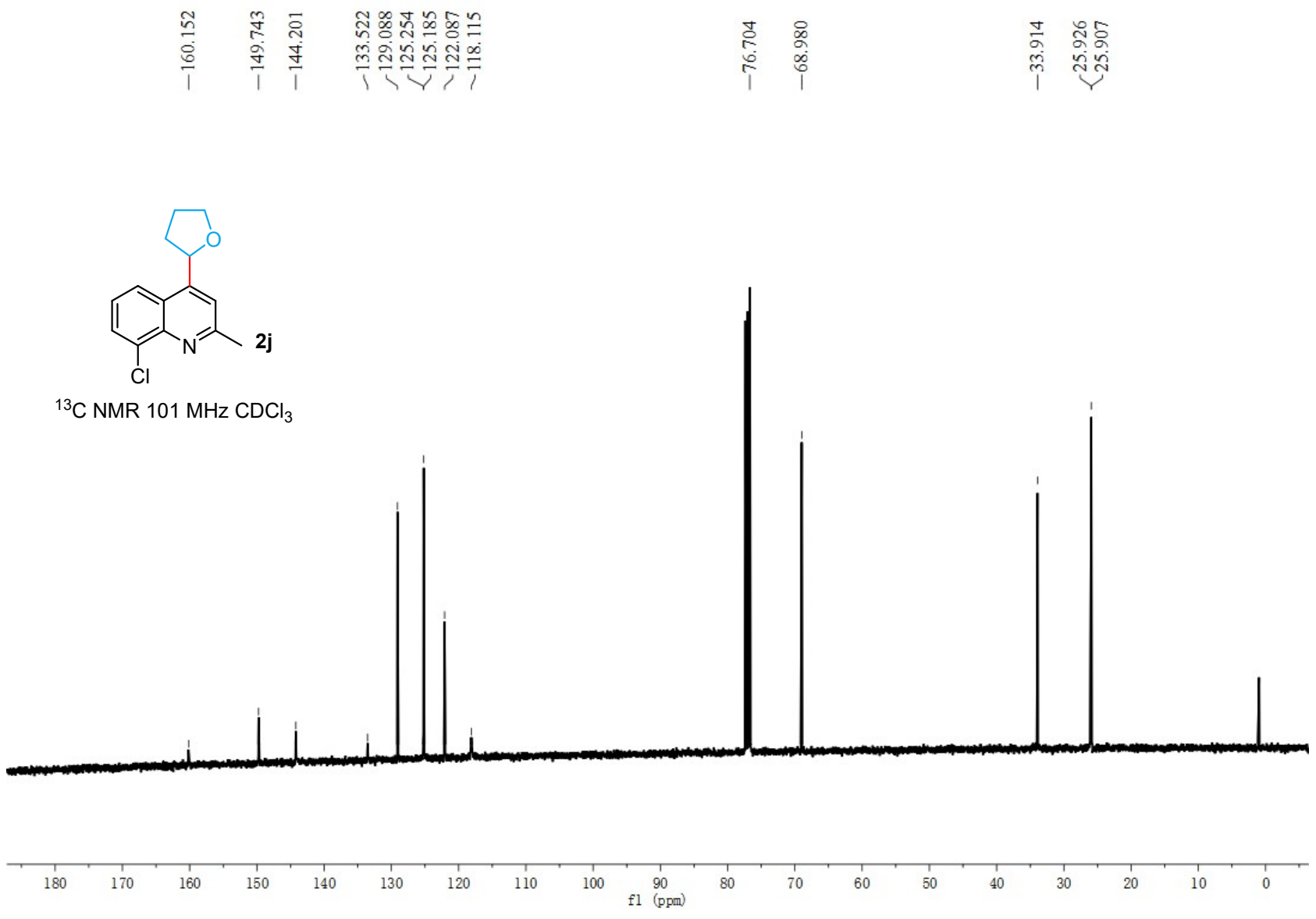
4.252
4.233
4.218
4.200
4.067
4.049
4.030
4.011

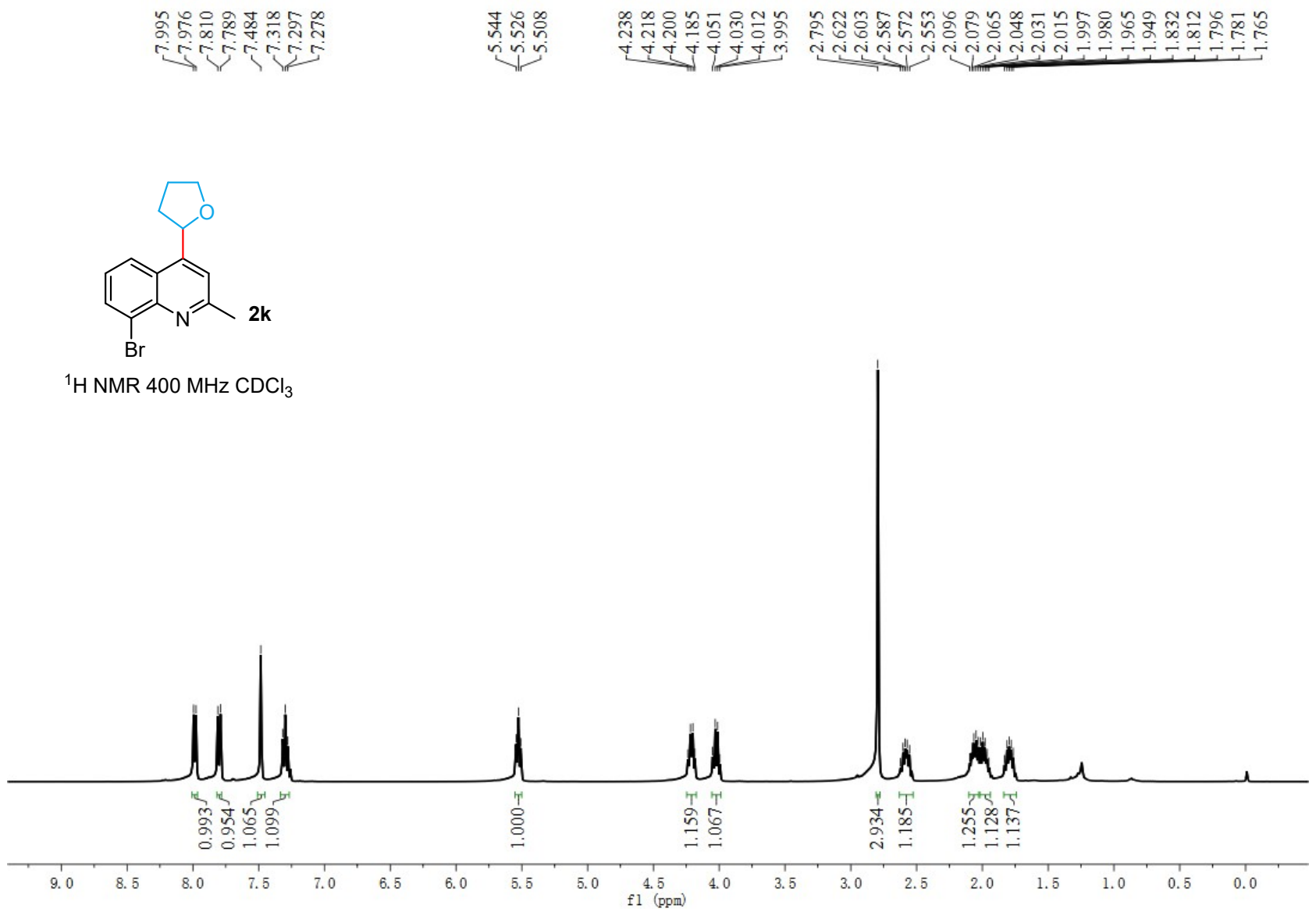
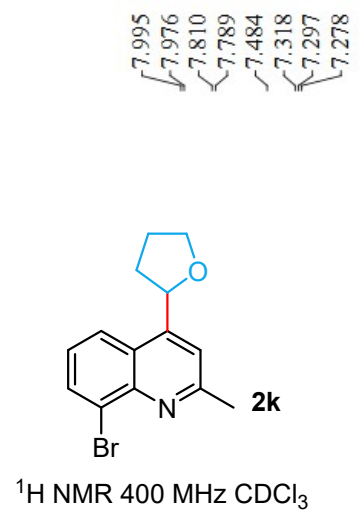
2.817
2.643
2.624
2.607
2.593
2.576
2.557
2.133
2.116
2.101
2.084
2.068
2.053
2.035
2.017
1.999
1.985
1.968
1.951
1.855
1.838
1.820
1.807
1.788
1.771

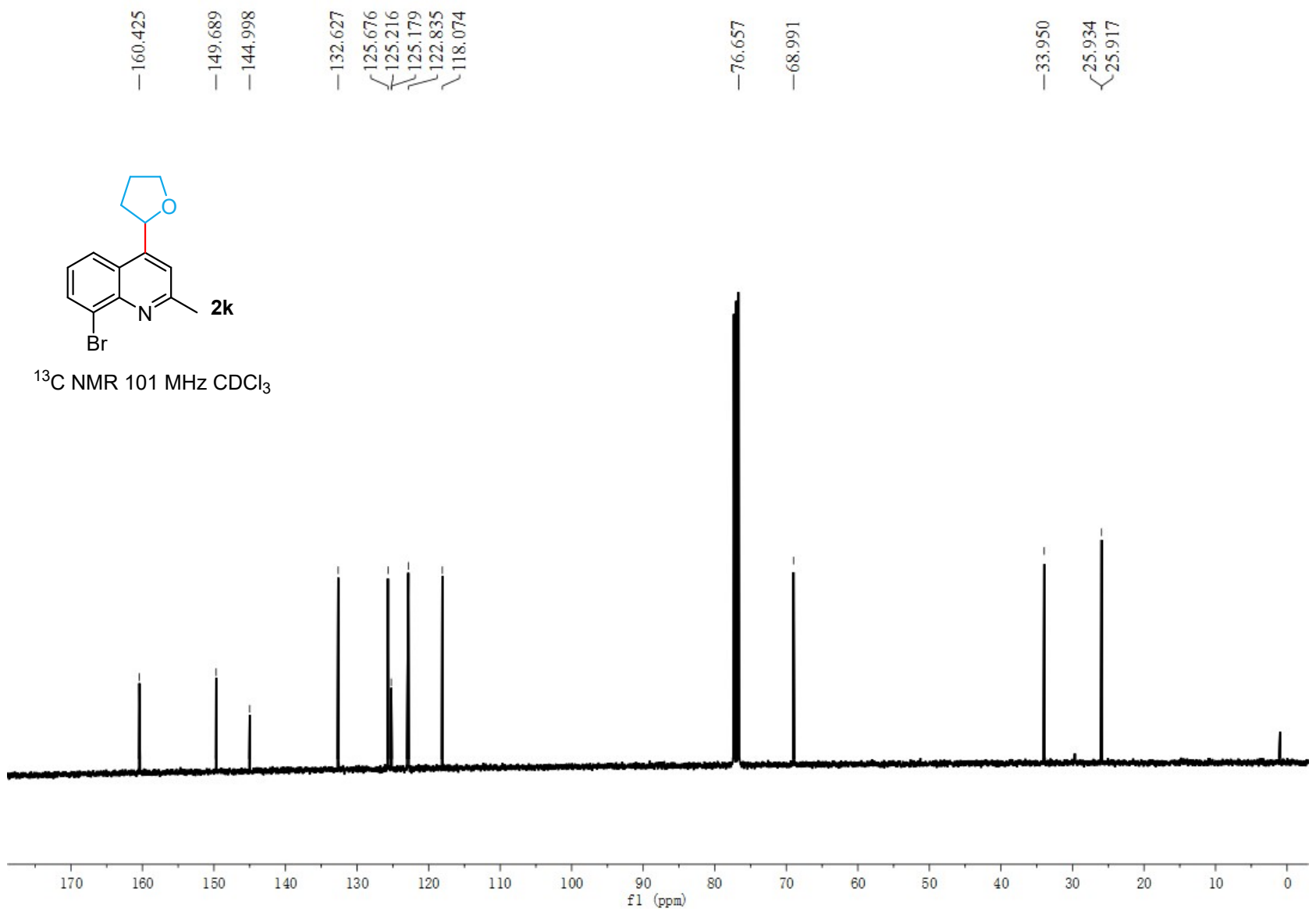
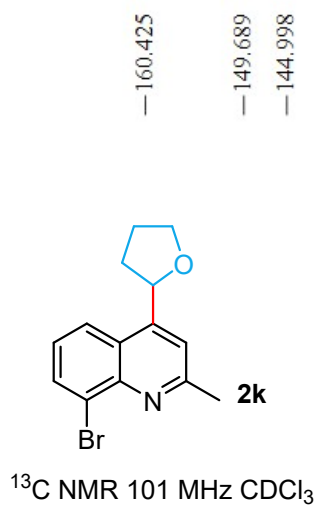


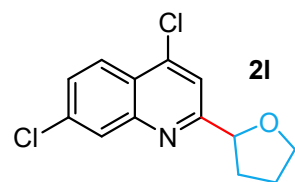
¹H NMR 400 MHz CDCl₃



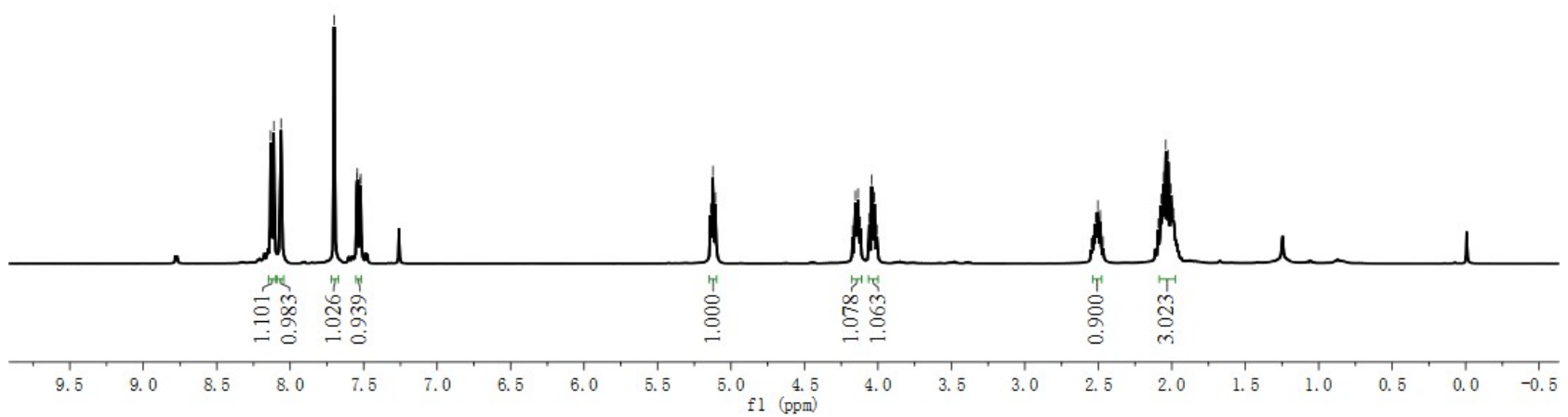








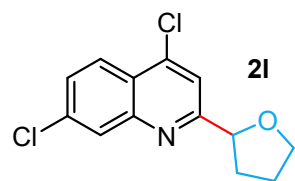
$^1\text{H NMR}$ 400 MHz CDCl_3



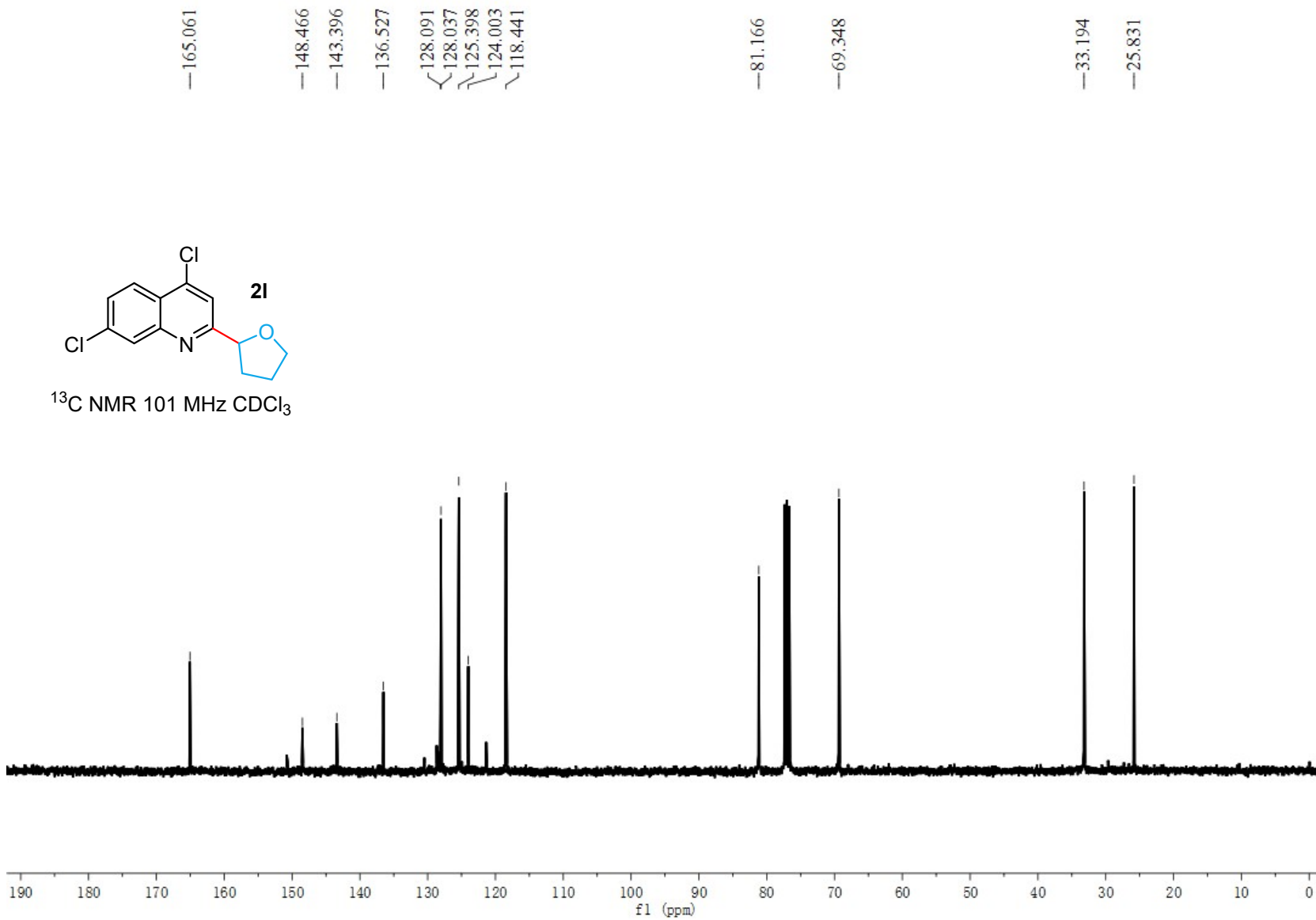
8.134
8.111
8.062
7.701
7.546
7.543
7.524
7.521

5.139
5.123
5.106
4.172
4.154
4.135
4.119
4.060
4.042
4.024
4.006

2.530
2.519
2.503
2.486
2.467
2.074
2.057
2.041
2.026
2.006
1.988

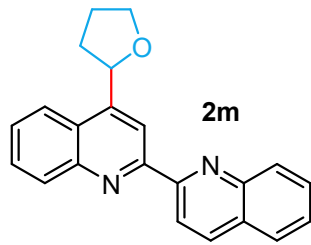


^{13}C NMR 101 MHz CDCl_3

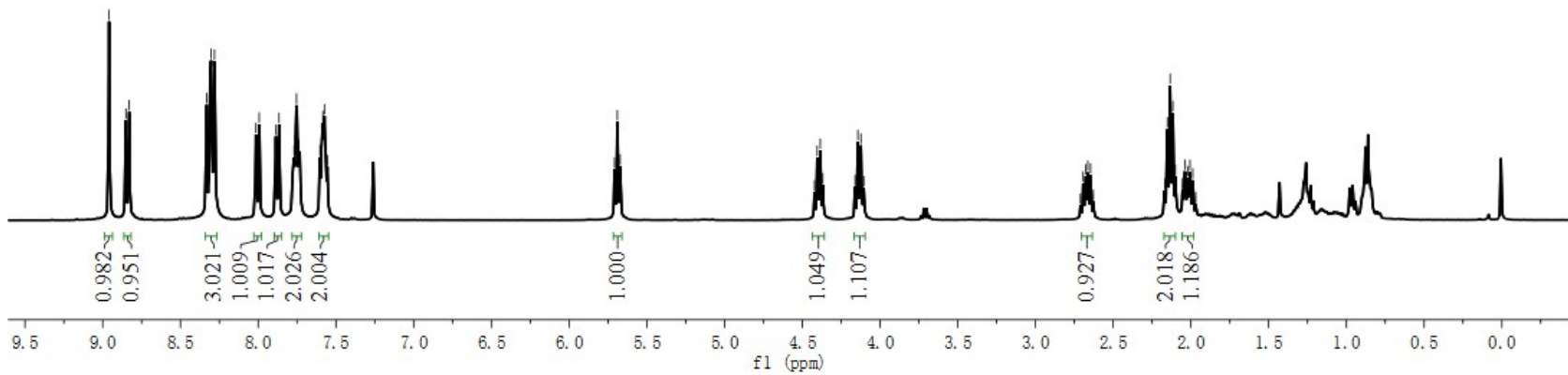


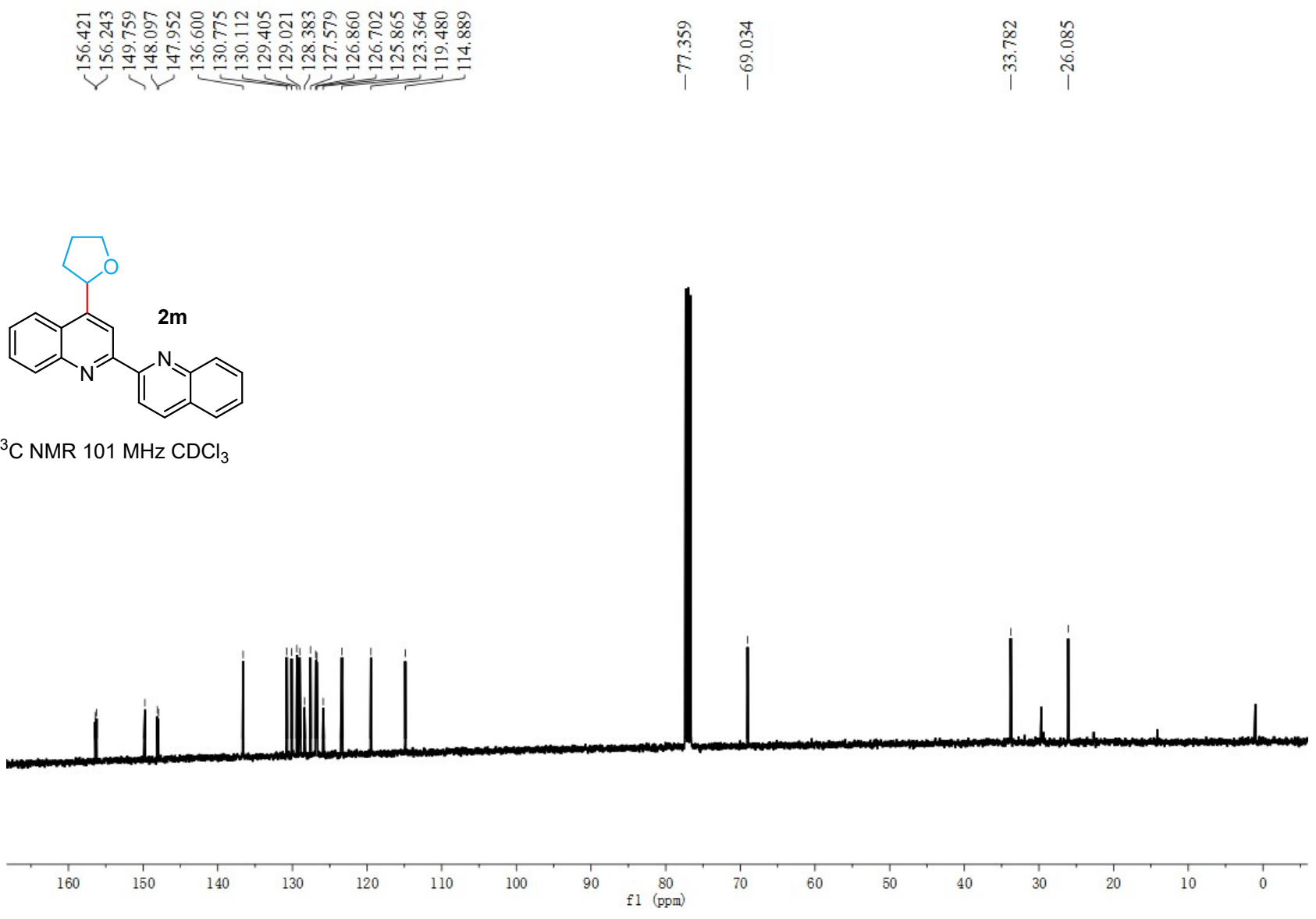
8.959
8.851
8.829
8.331
8.309
8.303
8.281
8.014
7.993
7.886
7.866
7.769
7.753
7.738
7.603
7.591
7.584
5.774
5.768
5.690
5.672

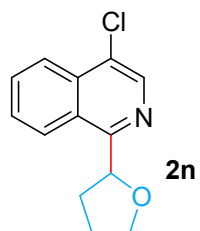
4.421
4.404
4.385
4.368
4.160
4.143
4.123
4.105
2.711
2.693
2.676
2.663
2.646
2.628
2.169
2.152
2.134
2.117
2.100
2.054
2.045
2.037
2.018
2.006
1.988
1.969



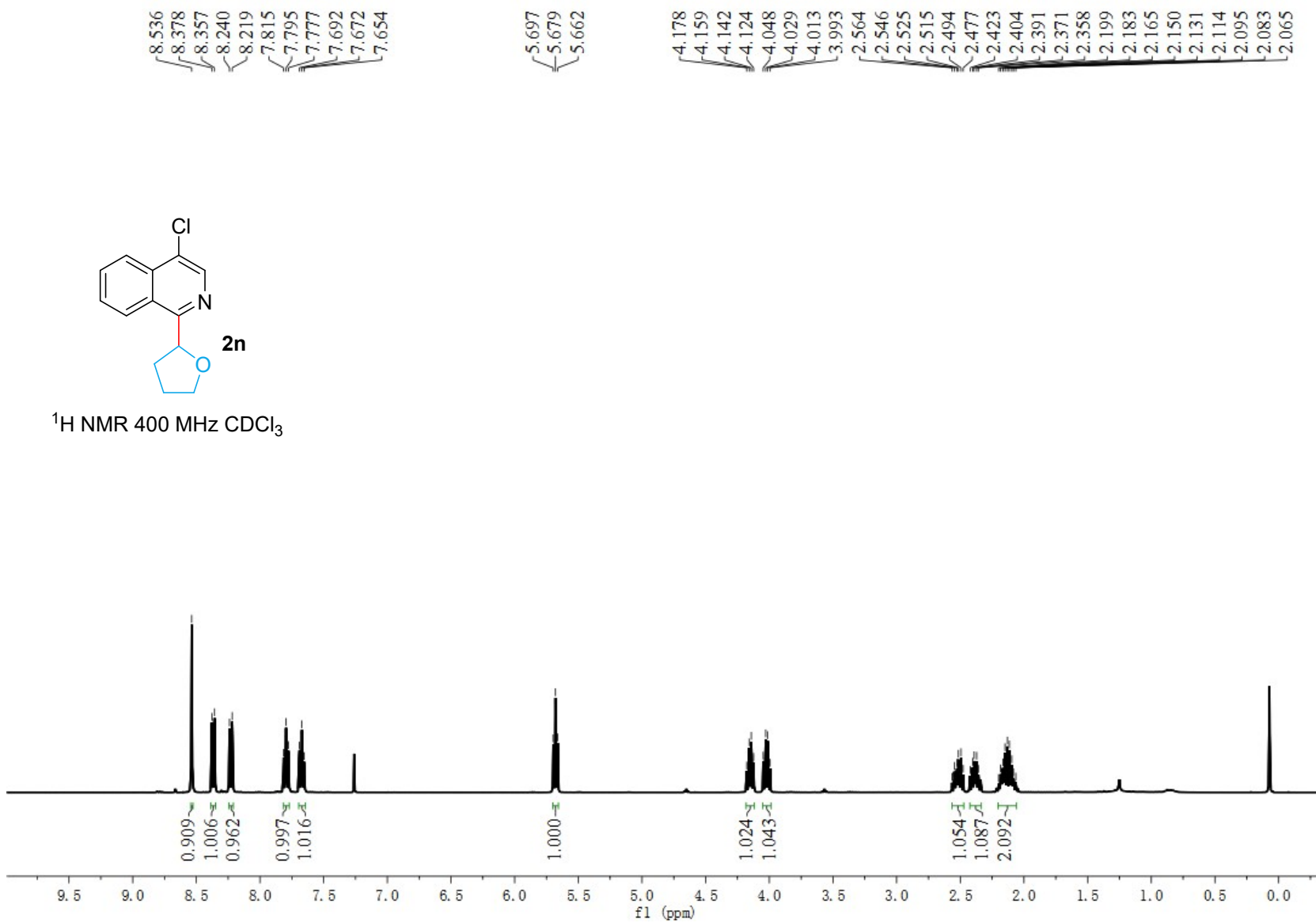
^1H NMR 400 MHz CDCl_3

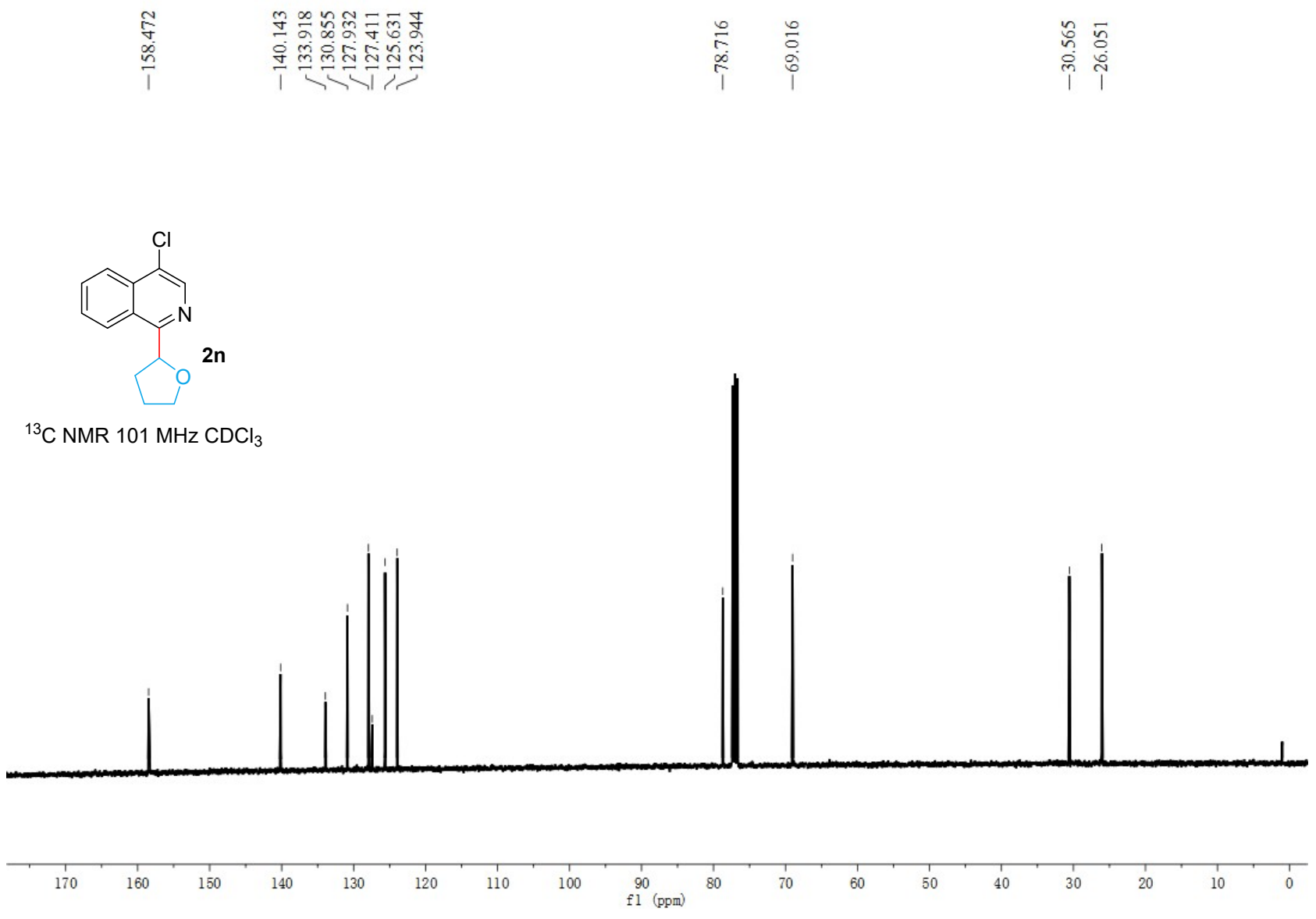






$^1\text{H NMR}$ 400 MHz CDCl_3

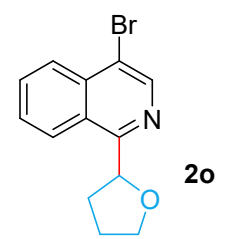




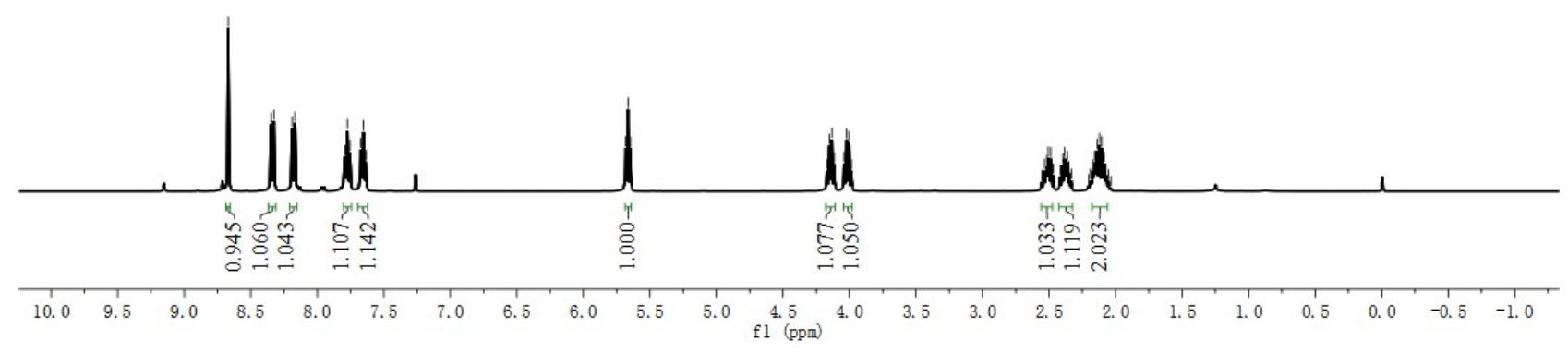
8.669
8.347
8.326
8.190
8.168
7.792
7.774
7.755
7.673
7.653
7.635

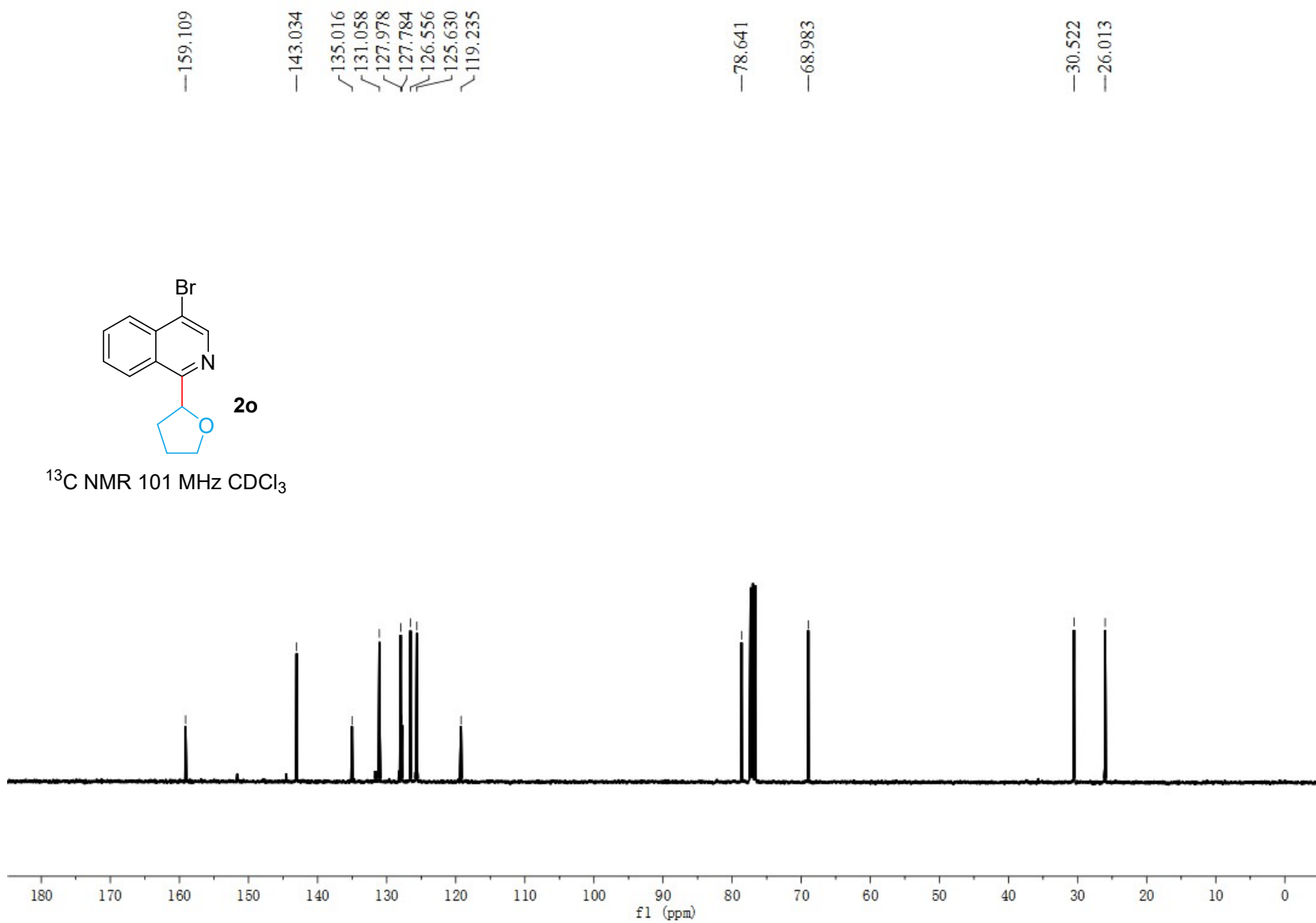
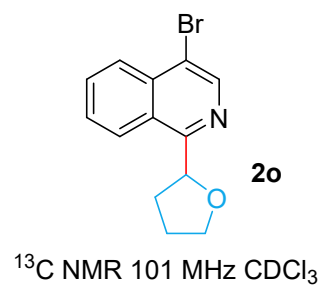
5.682
5.664
5.647

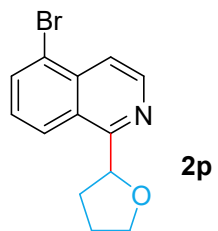
4.169
4.150
4.133
4.114
4.040
4.021
4.005
3.985
2.555
2.537
2.517
2.506
2.486
2.468
2.416
2.397
2.383
2.364
2.351
2.332
2.203
2.188
2.173
2.156
2.139
2.121
2.104
2.085
2.073
2.055
2.036



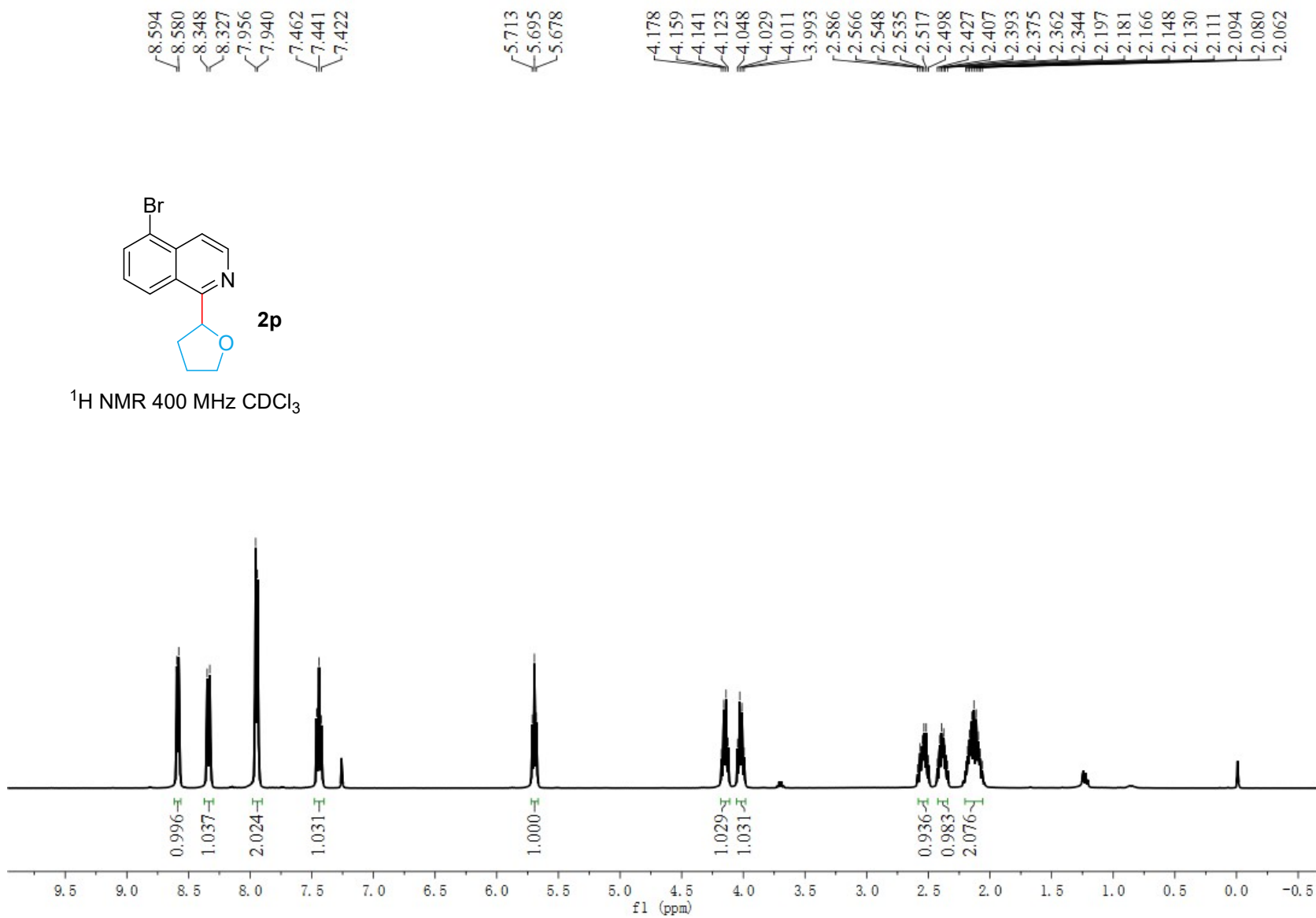
¹H NMR 400 MHz CDCl₃

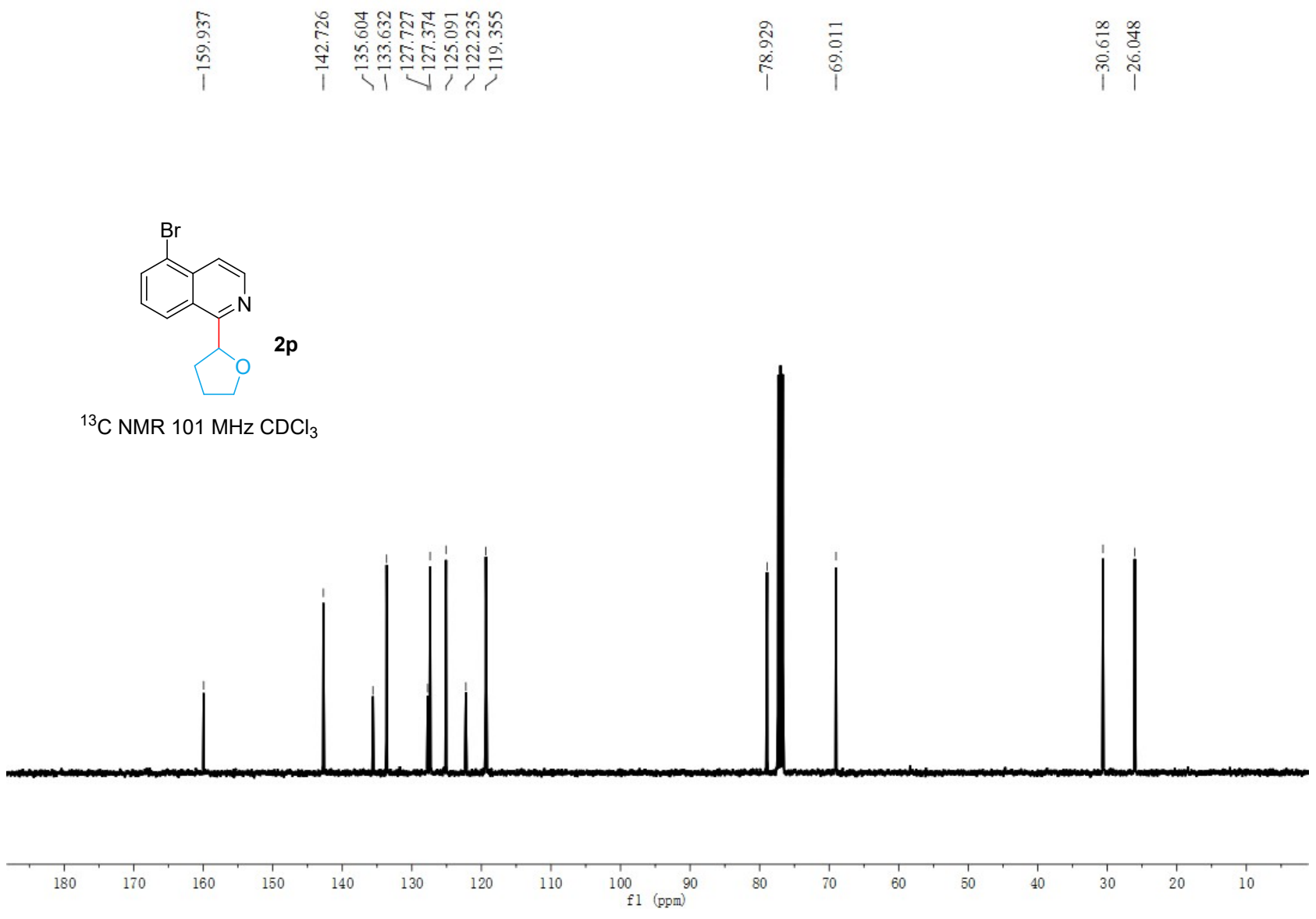




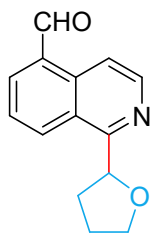


$^1\text{H NMR}$ 400 MHz CDCl_3



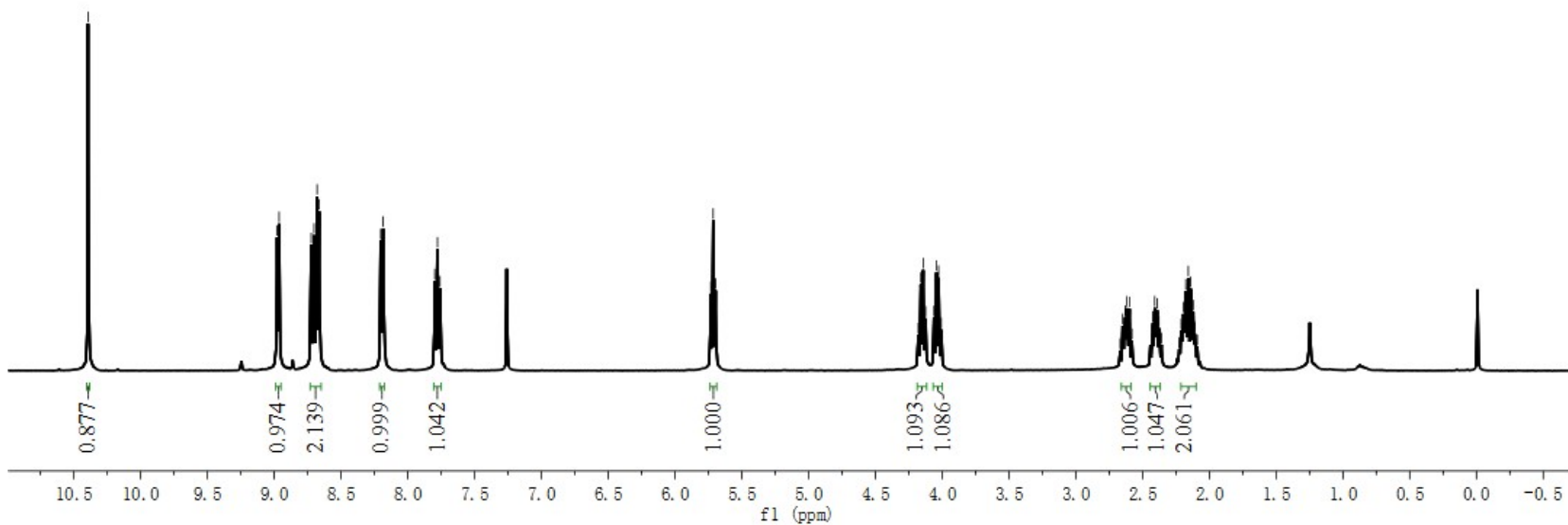


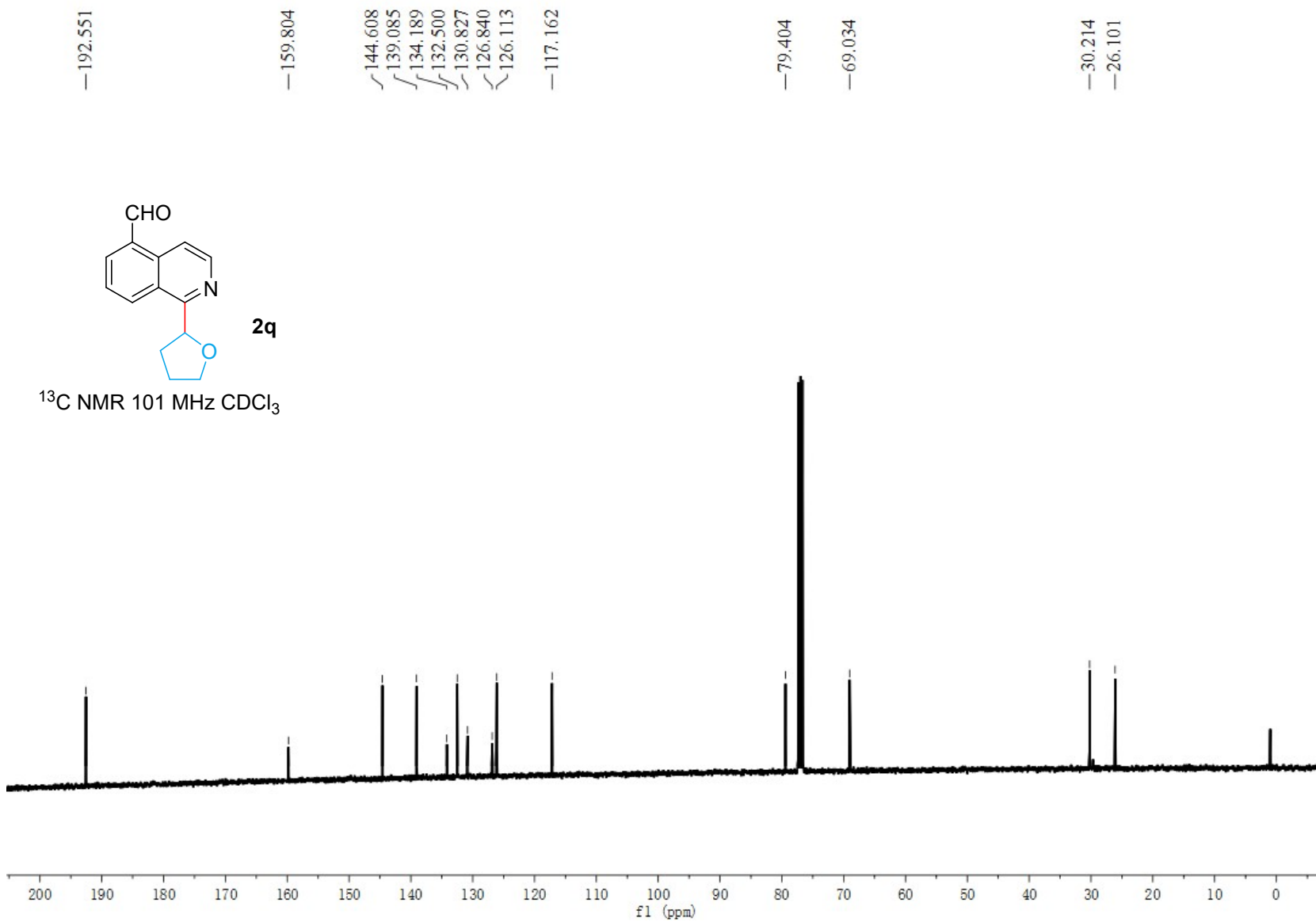
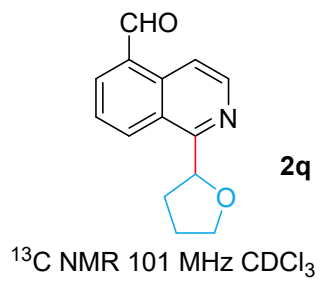
-10.394
 8.979
 8.964
 8.724
 8.702
 8.679
 8.664
 8.203
 8.185
 7.798
 7.778
 7.759
 5.732
 5.714
 5.697
 4.179
 4.160
 4.142
 4.124
 4.062
 4.042
 4.026
 4.007
 2.667
 2.649
 2.629
 2.617
 2.598
 2.579
 2.422
 2.409
 2.390
 2.377
 2.359
 2.225
 2.210
 2.194
 2.176
 2.157
 2.139
 2.121
 2.109
 2.090

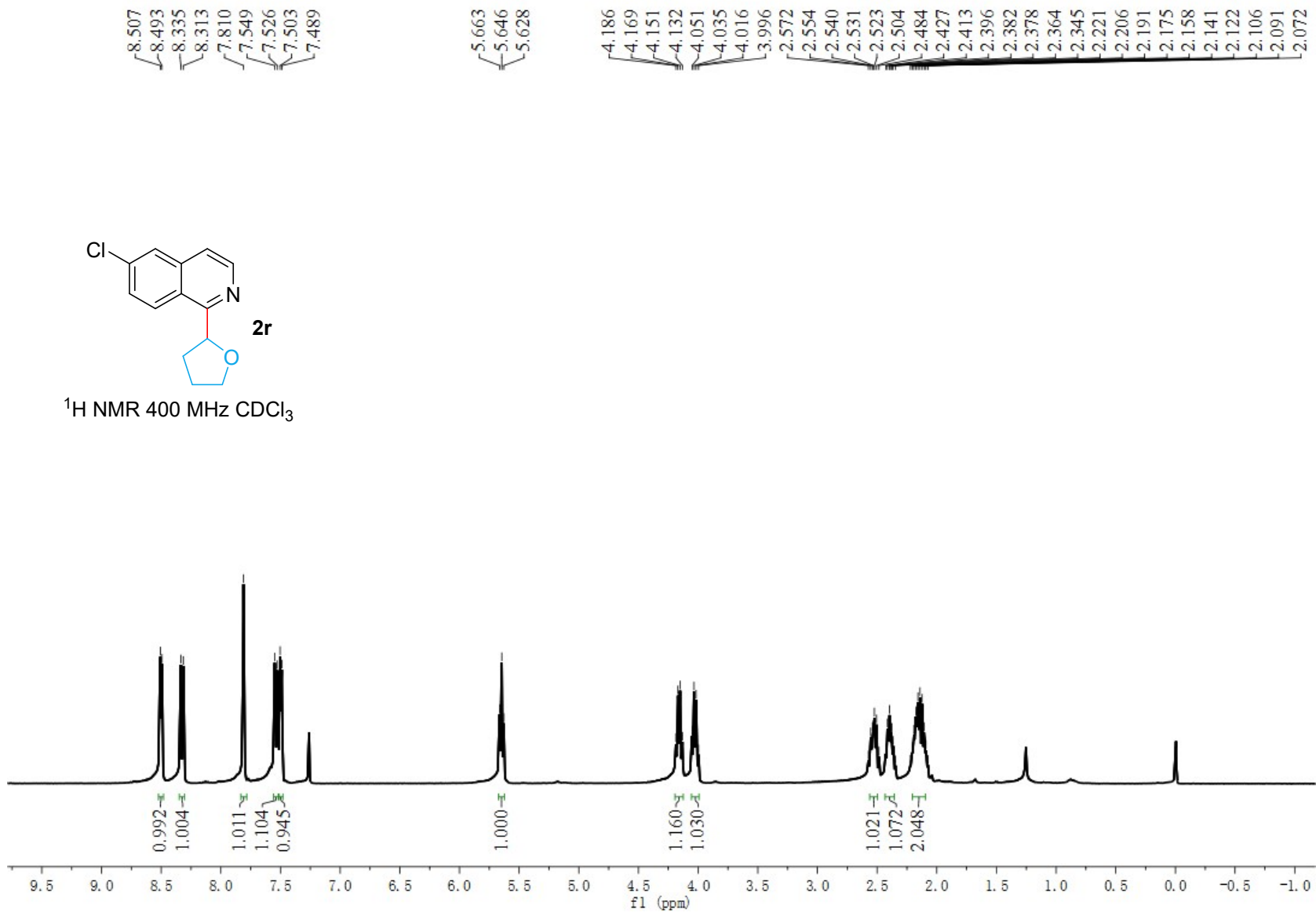
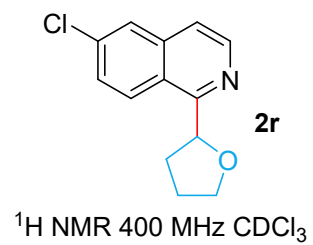


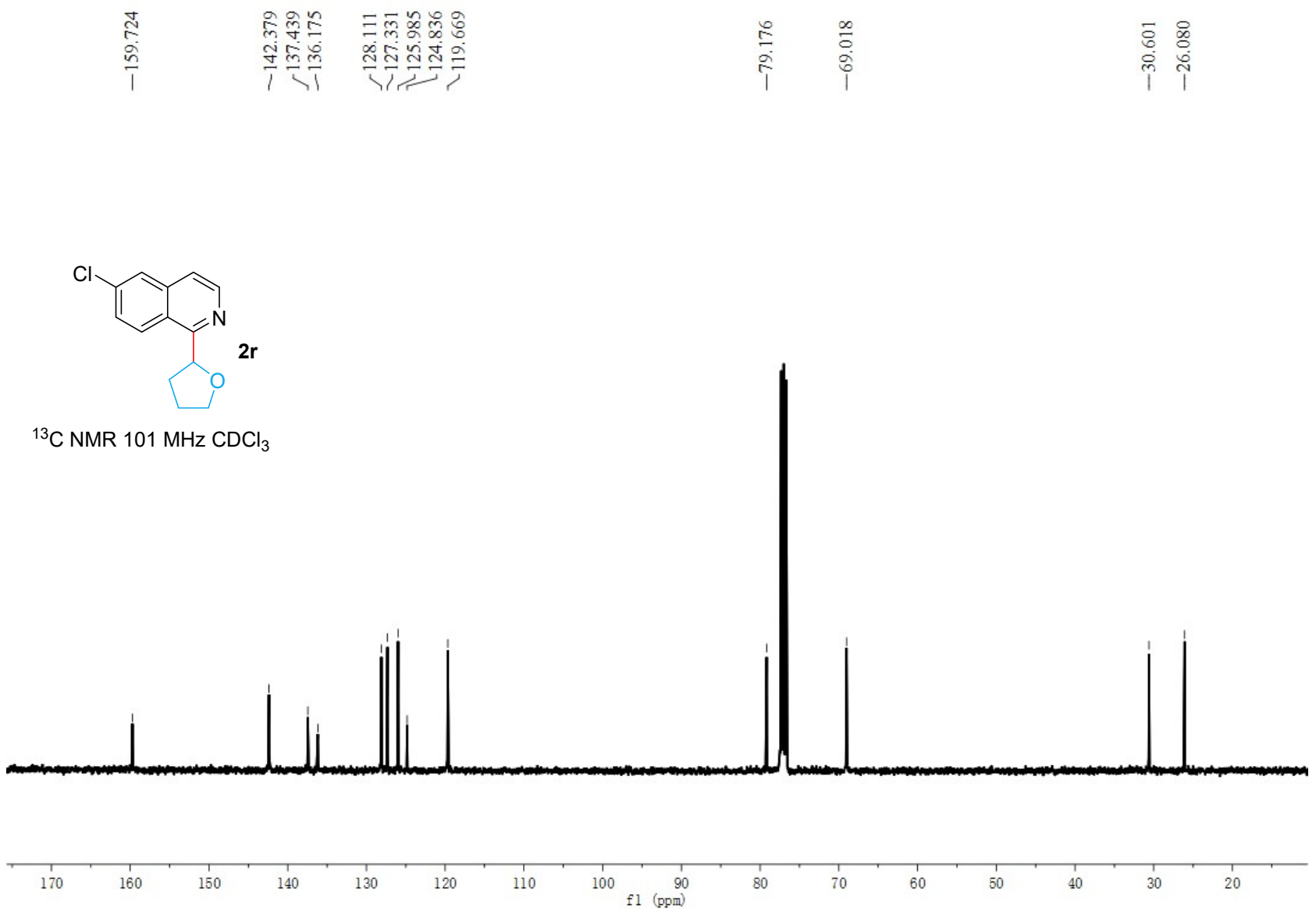
2q

¹H NMR 400 MHz CDCl₃





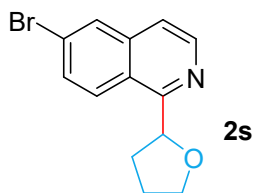




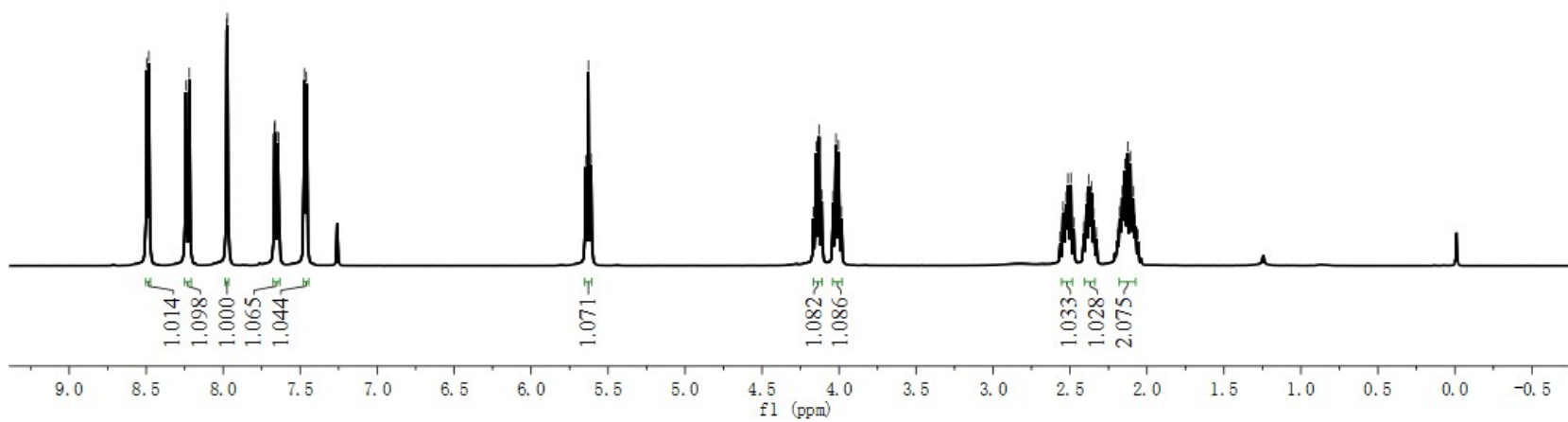
8.497
8.483
8.243
8.220
7.976
7.973
7.667
7.663
7.645
7.640
7.473
7.458

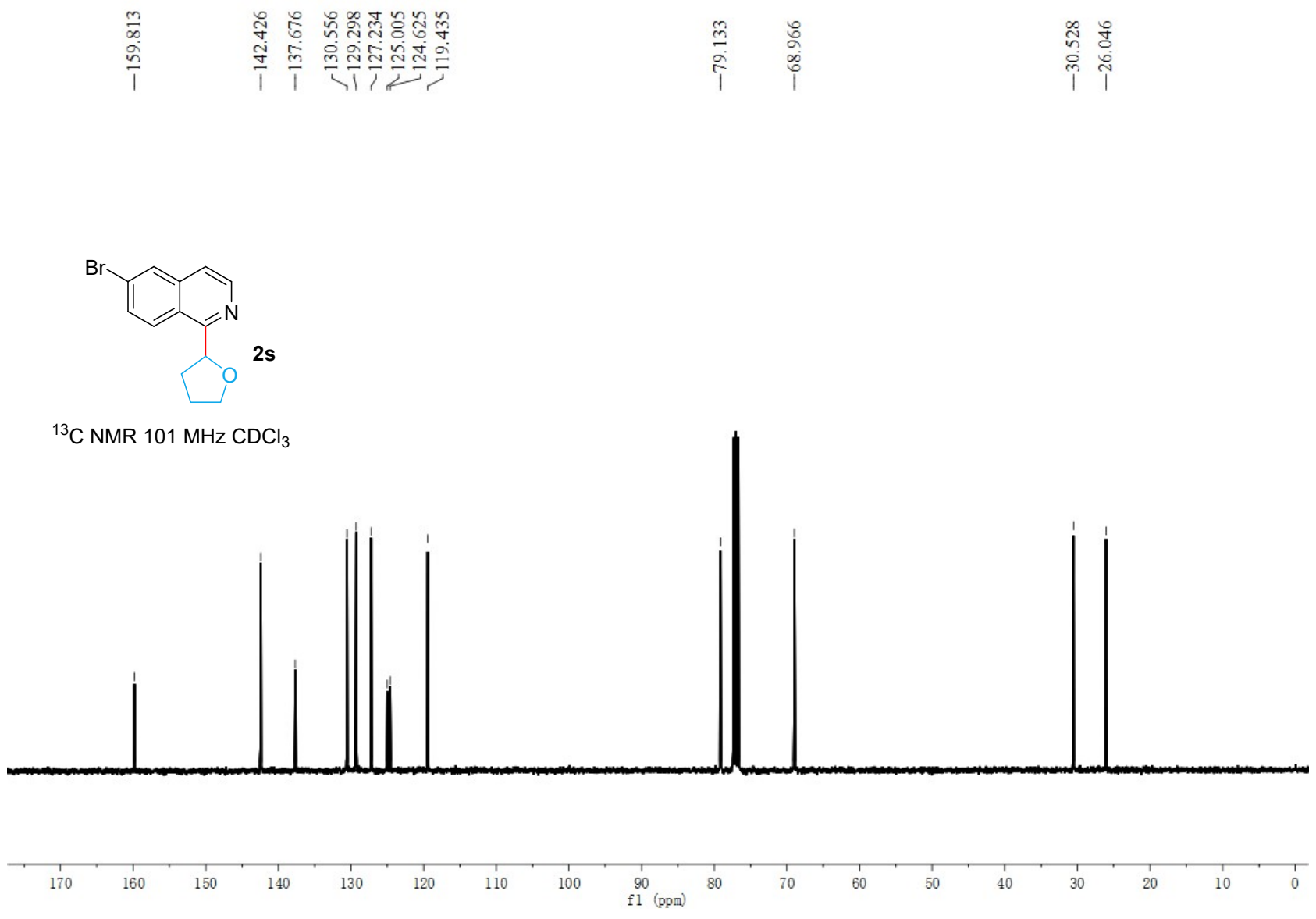
5.646
5.628
5.610

4.167
4.148
4.130
4.112
4.039
4.020
4.003
3.984
2.564
2.545
2.532
2.525
2.514
2.494
2.475
2.410
2.391
2.378
2.359
2.346
2.328
2.192
2.177
2.160
2.144
2.125
2.108
2.089
2.077
2.059



$^1\text{H NMR}$ 400 MHz CDCl_3

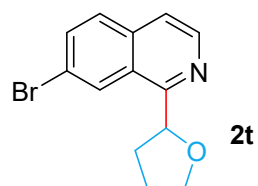




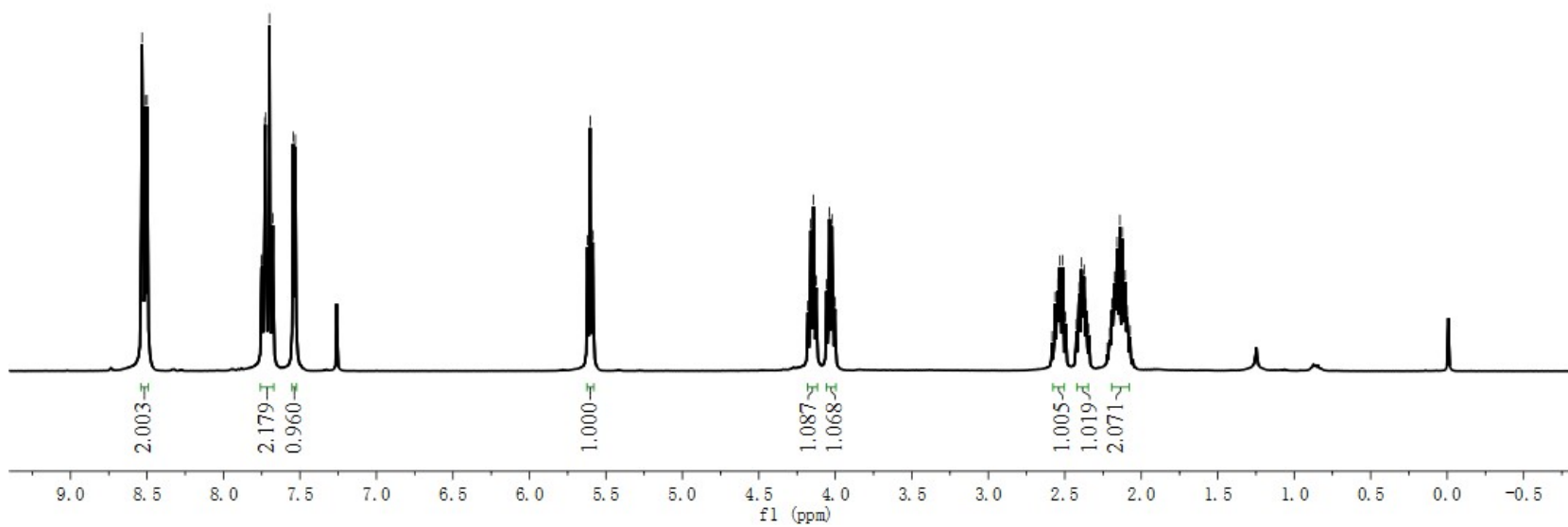
8.532
8.512
8.498
7.751
7.748
7.729
7.726
7.699
7.677
7.545
7.530

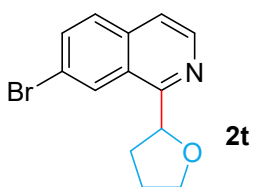
5.621
5.603
5.585

4.180
4.161
4.143
4.125
4.058
4.038
4.022
4.003
2.583
2.564
2.552
2.545
2.533
2.514
2.495
2.424
2.405
2.392
2.373
2.361
2.342
2.223
2.206
2.192
2.175
2.159
2.140
2.123
2.104
2.092
2.074

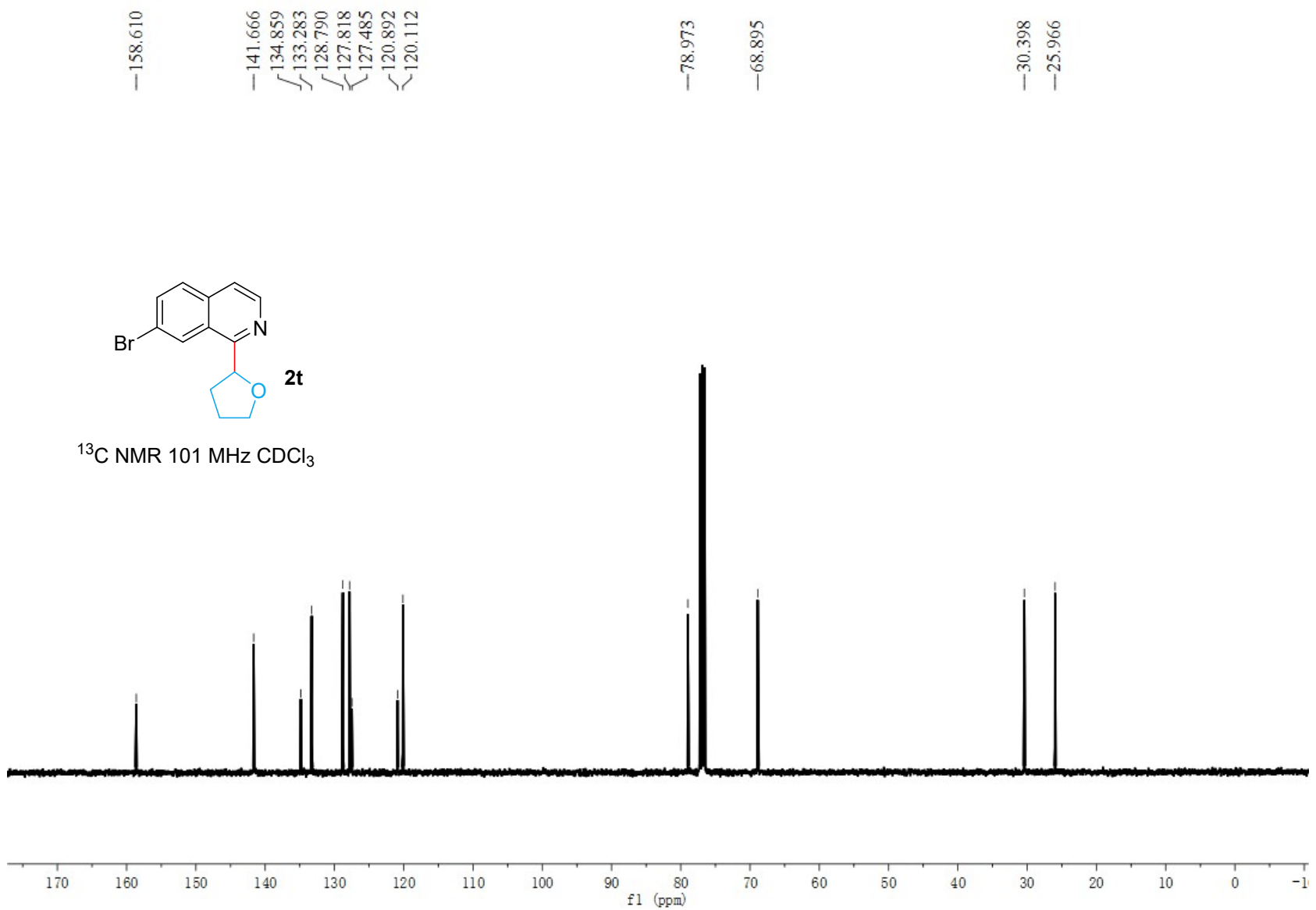


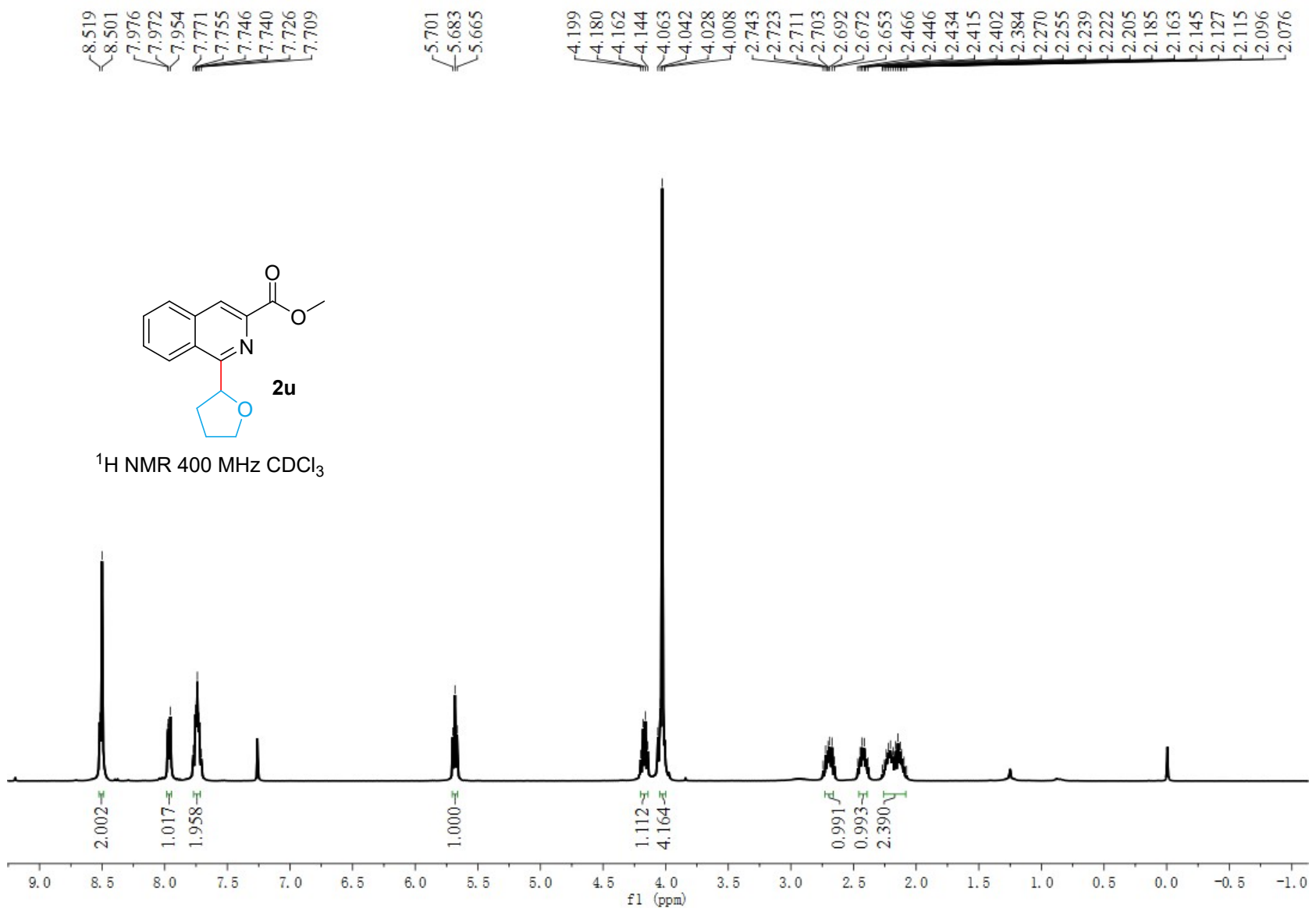
$^1\text{H NMR}$ 400 MHz CDCl_3

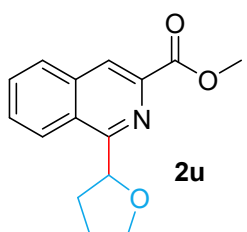




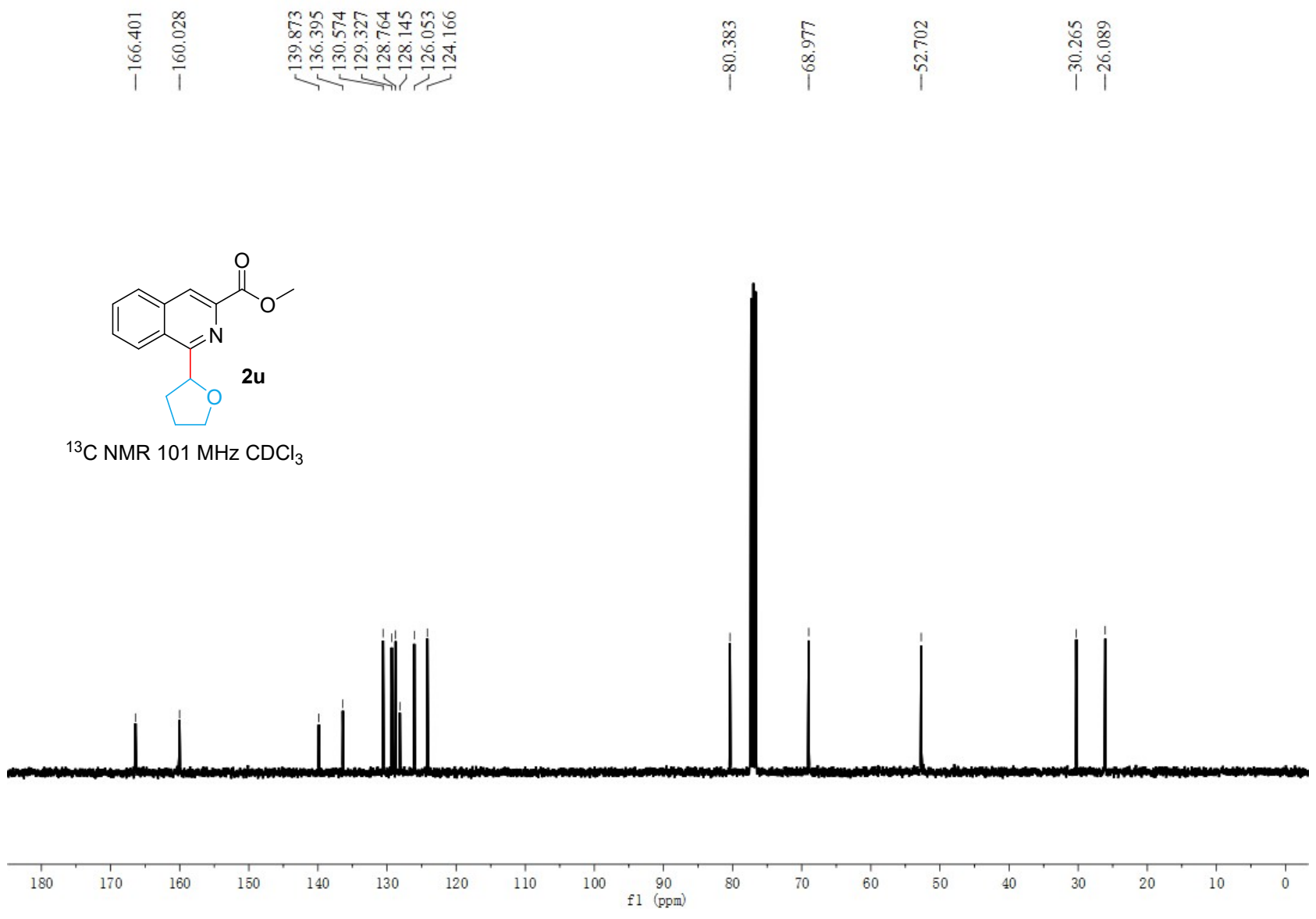
^{13}C NMR 101 MHz CDCl_3





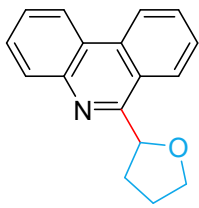


¹³C NMR 101 MHz CDCl₃



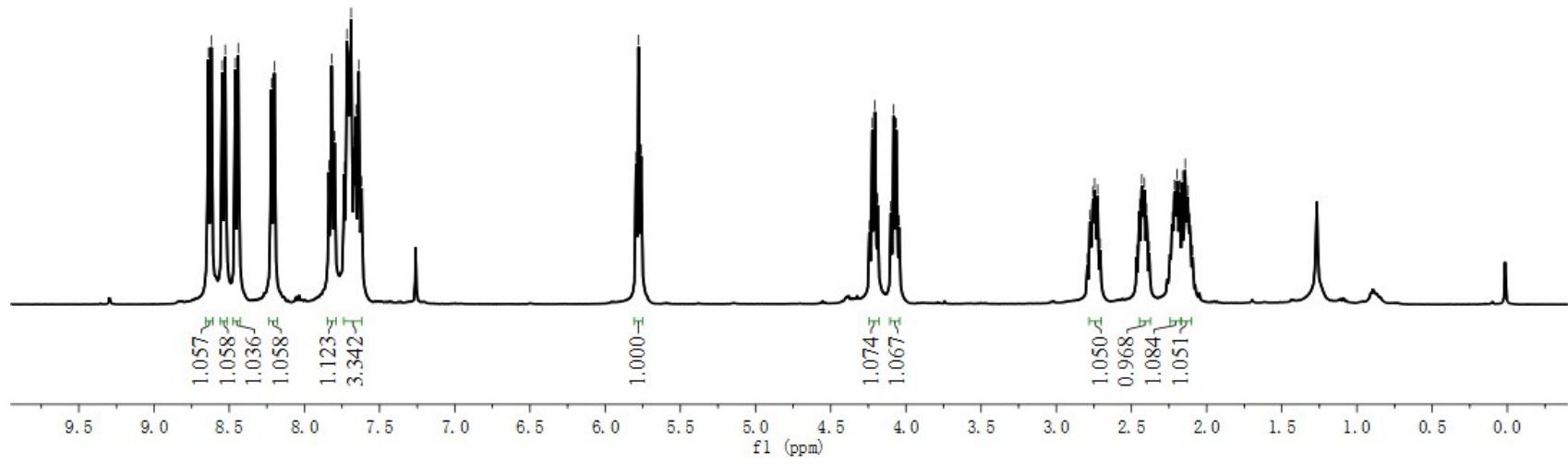
8.640
8.619
8.547
8.527
8.461
8.440
8.220
8.200
7.837
7.819
7.799
7.735
7.717
7.708
7.697
7.689
7.669
7.658
7.639
7.635
5.778
5.761

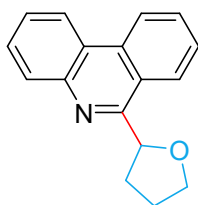
4.243
4.224
4.206
4.188
4.100
4.081
4.065
4.045
2.793
2.776
2.755
2.745
2.725
2.707
2.465
2.446
2.431
2.413
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2.213
2.196
2.178
2.161
2.142
2.125
2.113
2.094



2v

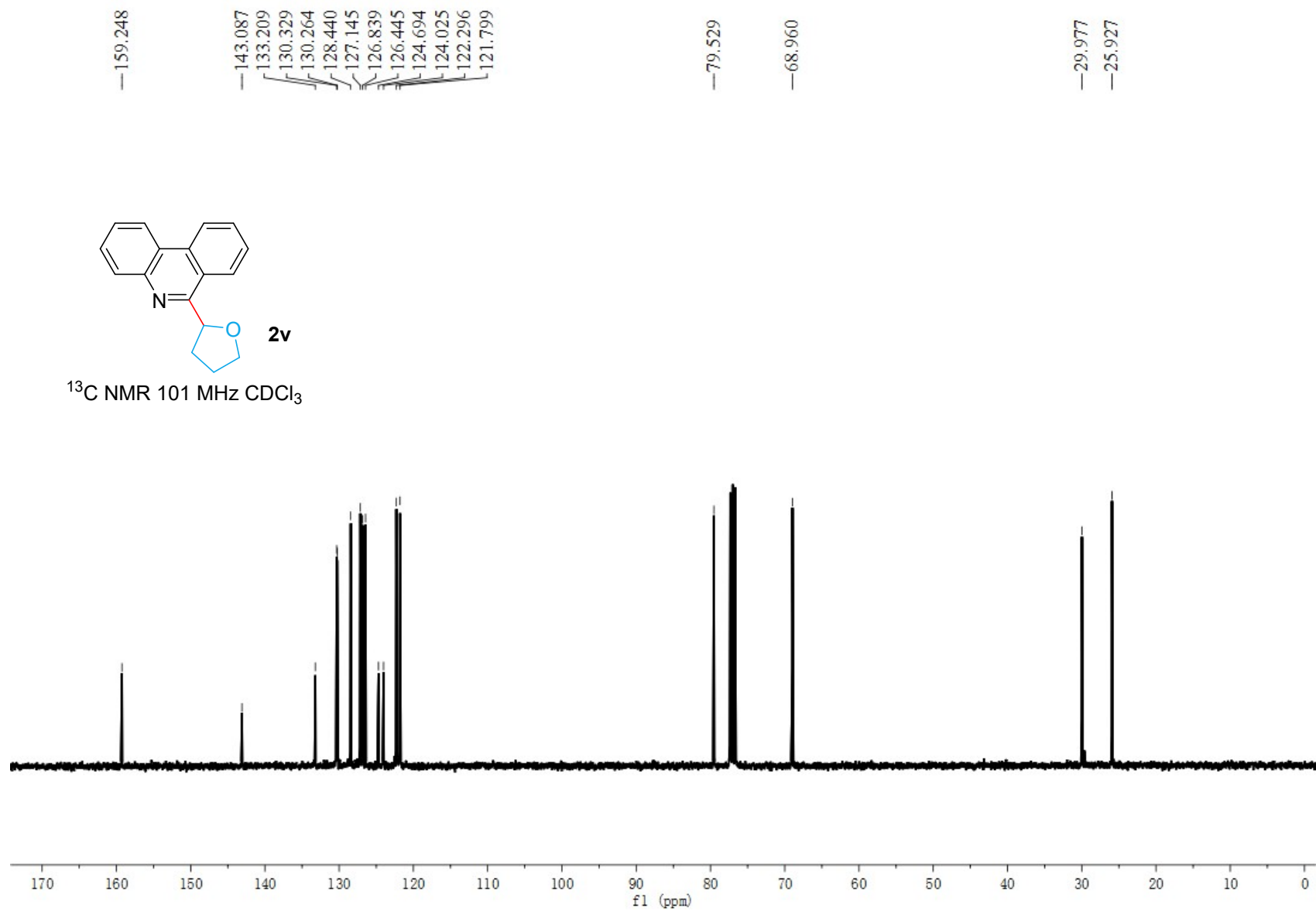
¹H NMR 400 MHz CDCl₃





2v

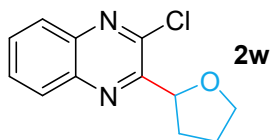
^{13}C NMR 101 MHz CDCl_3



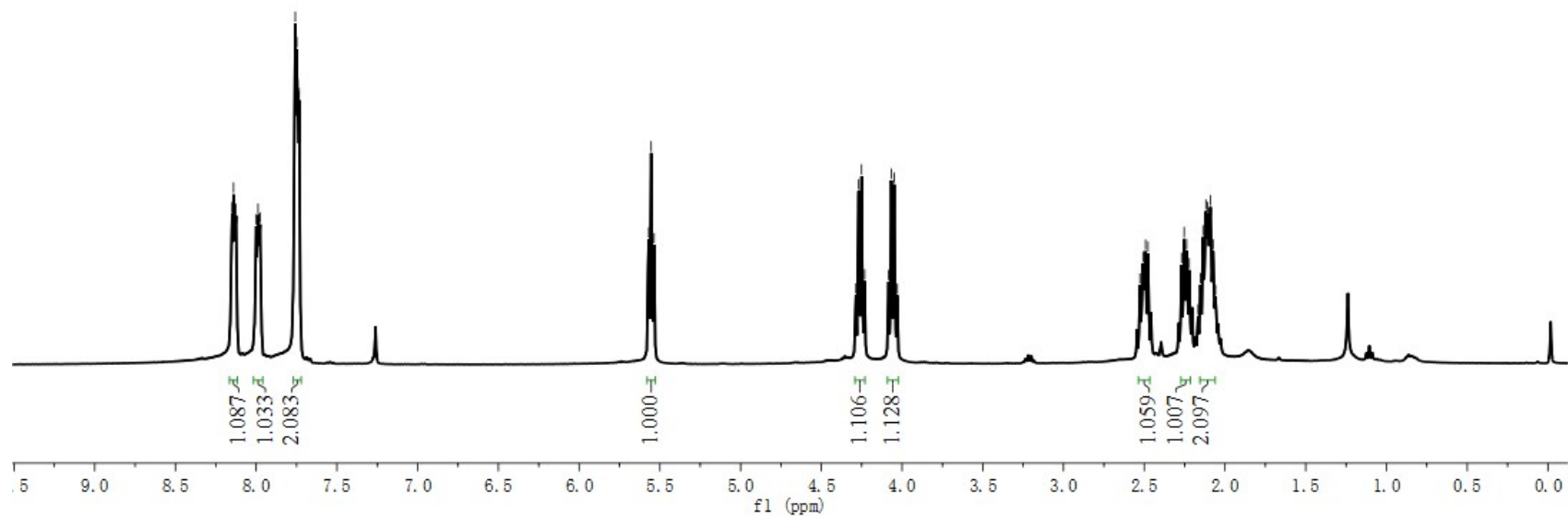
8.149
8.145
8.140
8.136
8.130
8.128
8.125
8.000
7.989
7.984
7.979
7.976
7.761
7.758
7.752
7.748
7.741
7.737
7.733

5.569
5.554
5.535

4.286
4.269
4.251
4.231
4.083
4.067
4.048
4.029
2.527
2.508
2.502
2.493
2.477
2.269
2.253
2.237
2.221
2.167
2.152
2.136
2.118
2.108
2.090
2.074
2.059



^1H NMR 400 MHz CDCl_3



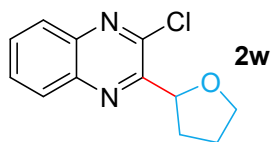
—154.472
—145.999
—141.299
—140.572
—130.580
—130.104
—129.257
—128.006

—77.973

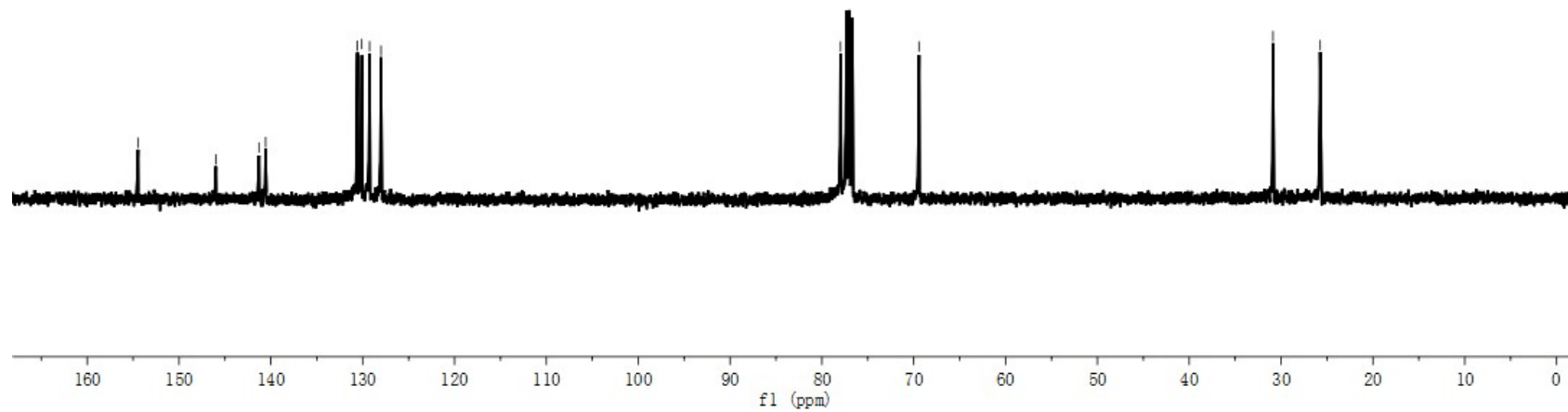
—69.425

—30.879

—25.738



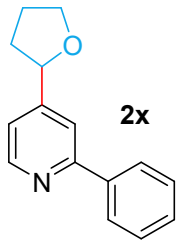
¹³C NMR 101 MHz CDCl₃



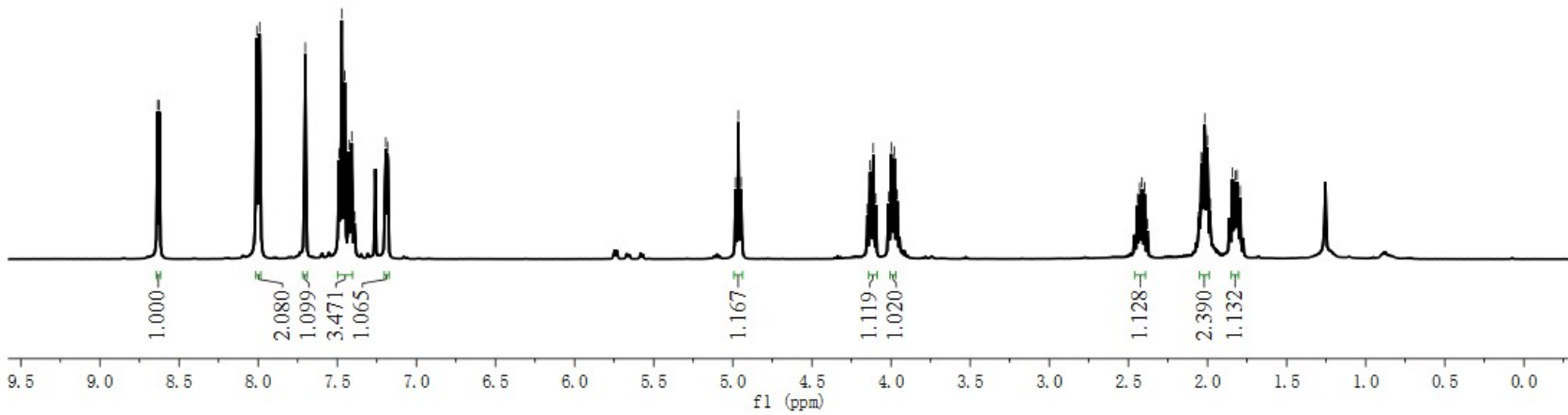
8.637
8.624
8.008
7.991
7.702
7.489
7.472
7.453
7.426
7.408
7.390
7.193
7.182

4.983
4.965
4.947
4.150
4.132
4.112
4.095
4.016
3.999
3.979
3.961

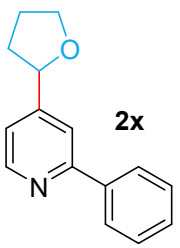
2.444
2.428
2.414
2.397
2.380
2.057
2.051
2.039
2.034
2.017
2.001
1.985
1.983
1.842
1.830
1.824
1.812
1.793



$^1\text{H NMR}$ 400 MHz CDCl_3



157.477
153.698
149.452
139.325
128.925
128.671
126.994
119.014
117.322
79.248
68.957
34.328
25.822



¹³C NMR 101 MHz CDCl₃

